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IRON ORES - REDOXIMETRY DETERMINATION OF TOTAL IRON - MERCURY POLLUTION
FREE METHODS
STATISTICAL EVALUATION OF INTERNATIONAL TEST DATA - 1984

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ISO/TC 102/SC 2 (WG 12-13)

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POLLUTION FREE METHODS

STATISTICAL EVALUATION OF INTERNATIONAL TEST DATA - 1984

by

R. Sutarno* and W.S. Bowman**

SUMMARY

The precision and accuracy of three analytical methods for the determination of total iron in iron ores by mercury pollution free redoximetry have been estimated from the data resulting from international tests conducted during the period of 1982/84. These international tests involved the participation of 35 laboratories from 10 countries using four iron ore samples containing 45 to 67% iron. It was found that these three methods have comparable accuracy with the following precision.

Precision	TiCl ₃ reduction WG 16B	Ag-reduction WG 17B	TiCl ₃ reduction Perchloric Acid Oxidation WG 23A
r %	0.156	0.209	0.203
P %	0.239	0.282	0.294
α_t %	0.055	0.074	0.073
α_L %	0.074	0.085	0.089

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In this table r is the repeatability, P is the permissible tolerance, σ_r is the within-laboratory standard deviation and σ_b is the between-laboratories standard deviation.

ISO-TC 102/SC 2 (WG 12-13)

Mars 1985

MINÉRAIS DE FER - DÉTERMINATION DU FER TOTAL PAR OXYDO-RÉDUCTION
 - MÉTHODES DE DOSAGE EN L'ABSENCE DE CONTAMINATION PAR LE MERCURE
 ÉVALUATION STATISTIQUE DE DONNÉES D'ESSAIS FAITS AU
 NIVEAU INTERNATIONAL - 1984

par

R. Sutarno* et W.S. Bowman**

RÉSUMÉ

On a évalué la précision et l'exactitude de trois méthodes analytiques servant à déterminer le fer total dans les minerais de fer par oxydo-réduction en l'absence de contamination par le mercure, à partir de données obtenues des résultats d'essais faits au niveau international entre 1982-1984. Trente-cinq laboratoires provenant de dix pays ont participé à ces essais effectués à partir de 4 échantillons de minerai de fer contenant de 45 à 67 % de fer. On a constaté que ces trois méthodes d'analyse ont une exactitude comparable à la précision indiquée au tableau suivant.

Précision	Réduction TiCl_3 WG 16B	Réduction Ag WG 17B	Réduction TiCl_3 Oxydation à l'acide perchlorique WG 23A
r %	0,156	0,209	0,203
P %	0,239	0,282	0,294
σ_r %	0,055	0,074	0,073
σ_L %	0,074	0,085	0,089

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Dans ce tableau, la répétabilité est représentée par r , l'écart admissible par P , l'écart type en laboratoire par σ_r et l'écart type entre les laboratoires par σ_L .

INTRODUCTION

In recent years, ISO/TC 102/SC 2 (International Organization for Standardization, Technical Committee 102-Iron Ores, Subcommittee 2-Chemical Analysis) has been working on the development of methods for the determination of total iron content in iron ores as possible replacements for the existing ISO standard method ISO 2597. The primary reason for replacing this standard is to avoid the use of mercuric chloride and pollution problems associated with the disposal of wastes containing mercury. Two titrimetric methods using Ti^{+3} and Ag as reductors have been under investigation since 1976. These methods have been found to be suitable as international standards. However, in view of the reported shortage of sodium peroxide as the fusing agent, the subcommittee decided, at the eleventh meeting, that the working groups responsible for developing these methods (WG 16B and WG 17B) find an alternative method for sample decomposition¹. Meanwhile, at the same meeting, USA proposed another method for the same purpose. This method is a modification of Ti^{+3} reduction method with the use of perchloric acid to oxidize the excess of Ti^{+3} . A new working group (WG 23A) was formed to undertake this development. The three working groups conducted international tests simultaneously using the same set of test samples. In addition, WG 23A also used sample 76-17 as one of the test samples. This sample is known to contain vanadium; it was used to investigate the degree of interference of this element.

The purpose of this report is to present the results of the statistical evaluation of the analytical data resulting from the international tests conducted by the three WG's, during the period of 1982/84.

PRINCIPLE OF ANALYTICAL METHOD

1. Decomposition of test sample

This part of the analytical method is common for all three methods. The test sample was decomposed by treatment with hydrochloric and sulphuric acids. The undissolved portion was filtered from the main solution and fused with potassium pyrosulphate. The melt was dissolved in hydrochloric

acid and the iron was separated by precipitation with ammonia solution and redissolved with hydrochloric acid. The solution was then combined with the main solution and ready for reduction.

2. Reduction

(a) Ti³⁺ - reduction (WG 16B)²

The major portion of the trivalent iron was reduced by stannous chloride and the remainder by titanium trichloride using indigo carmine indicator. The excess of titanium trichloride was oxidized with dilute potassium dichromate solution. The reduced iron was titrated with potassium dichromate solution using sodium diphenylamine sulphonate indicator.

(b) Ag - reduction (WG 17B)³

The solution was passed through a silver reductor. A mixture of phosphoric and sulphuric acids was added to the solution. The total iron was then titrated with potassium dichromate solution using sodium diphenylamine sulphonate indicator.

(c) Ti³⁺ - reduction using perchloric acid (WG 23A)⁴

Most of the iron was reduced in a hot 6 M hydrochloric acid medium with stannous chloride, followed by the addition of a slight excess of Ti³⁺ solution. The excess Ti³⁺ was oxidized in the hot solution by perchloric acid. The solution was rapidly cooled and the reduced iron was titrated with potassium dichromate solution using sodium diphenylamine sulphonate indicator.

STATISTICAL ANALYSIS

The analytical data collected from the three working groups (from here on designated by the working group numbers WG 16B, WG 17B and WG 23A) during the period of 1982/84 were statistically evaluated by the procedure described in document ISO/TC 102/SC 2 N 768E⁵. This procedure is schematically illustrated in Figure 1. The raw data are also plotted in Figures 2. The boxed points in these figures signify that these data were

suspected outliers and were not used to estimate the grand mean and precision. The letters above the boxes signify the cause of the suspected data i.e. C - by Cochran's test and D - by Dixon's test. The detailed computations are appended in this report and are summarized in Table 1. Table 2 lists the outlying data.

Comparison with previous international test

The results of the statistical evaluation of the present international test data are compared with those obtained from the previous test (1980/82)⁶. Table 3 shows the comparison of the means and precision obtained from the two sets of international tests.

Comparison between methods

Comparison is also made between the results obtained by different methods. Table 4a shows these comparisons both in terms of grand mean, and precision. Table 4b shows the same comparison in relative terms.

OBSERVATIONS

Table 1 shows the overall means and precision obtained by the three methods. In all cases these results show equal or better precision than that of the existing ISO 2597¹ shown in Table 6. The α_L/α_T ratios are below 2, the bench mark value for methods developed by the subcommittee. The number of outliers is generally acceptable with the exception of a few laboratories (GER 1 and FR 1) for WG 16B data. These laboratories seem to have difficulty in applying the method. Tables 3 show that the differences between the results of the present international tests and those of the previous tests (for WG 16B and WB 17B) are not statistically significant. The only difference between these two sets of tests was the method of decomposition of the test samples, in the 1980/82 test the samples were decomposed by fusion using sodium peroxide. This indicates that the two methods of decomposition produce practically the same results; they both can be used as alternative methods within the standard. In all cases, the regression lines between precision and the level of concentration show virtually zero slopes. Therefore it is recommended that, for simplicity,

the precisions of these methods be expressed as constants. These recommended precisions are listed in Table 5. Both Tables 4 and 5 show that in terms of both accuracy and precision, these three methods are comparable. Indeed, at the eleventh meeting, they were all accepted as ISO standard methods to replace the existing ISO 2597.

REFERENCES

1. "Final report of Eleventh meeting of ISO/TC 102/SC 2 - Iron Ores/ Chemical Analysis, Ottawa 20, 21, 23, 24 September 1982", ISO/TC 102/ SC 2 N 735E, 1985-05-23.
2. "Determination of total iron content - Titanium(III) chloride reduction method - acid decomposition", Convener's report ISO/TC 102/SC 2/WG 16B N 760E, August 1984.
3. "Iron ores - Redoximetry determination of total iron - Mercury pollution free method", Convener's report ISO/TC 102/SC 2/WG 17B N 757E, July 1984.
4. "Iron ores - Determination of total iron content - Pollution free titrimetric method (perchloric acid oxidation)", Convener's report ISO/TC 102/SC 2/WG 23A N 754E, March 1984.
5. "Procedures for statistical evaluation of analytical data resulting from international tests", ISO/TC 102/SC 2 N 768E, September 1984.
6. "Iron ores - Determination of Fe-total by non mercury titrimetric methods - Statistical evaluation of international test data - 1982", R. Sutarno and W.S. Bowman, MRP/MSL 82-78 (IR), July 1982.
7. "Iron ores - Determination of total iron content - Titrimetric methods", ISO 2597, 1973.
8. "Results of international tests on eight iron ore samples - Determination of total iron - Document 102/SC 2 N 130E", ISO/TC 102/SC 2 N 147E, May 1968.

Table 1a

190/TC 102/SC 2/NG 188 - FE-TOTAL TICL3 REDUCTION ACID DISSOLUTION

SUMMARY OF PRECISIONS FOR ALL SAMPLES

ELEMENT	SAMPLE	K0	K	NO	N	MEAN(X)	REPEATABILITY	PERMISSIBLE TOLERANCE	SIGMA-R	SIGMA-L	SIGMA-L/SIGMA-R
IRON	83-2	17	15	51	45	66.7585	.1522	.2668	.05379	.08628	1.60
IRON	78-12	17	15	51	45	60.6304	.1857	.2724	.05855	.08691	1.48
IRON	81-2	17	14	51	42	59.5597	.1213	.2093	.04284	.08745	1.57
IRON	77-8	18	14	48	42	45.8803	.1849	.2058	.06532	.05617	.86

THE REGRESSION EQUATIONS ARE:

CORR. COEFF.

R	=	-.0018	X	+	.2589	-.5819
P	=	.0030	X	+	.0686	.7239
SIGMA-R	=	-.0008	X	+	.0915	-.5818
SIGMA-L	=	.0015	X	+	-.0126	.8738

Table 1b

ISO/TC 102/SC 2/MG 178 - FE-TOTAL AG REDUCTION ACID DISSOLUTION

SUMMARY OF PRECISIONS FOR ALL SAMPLES

ELEMENT	SAMPLE	K0	K	NO	N	MEAN(X)	REPEATABILITY	PERMISSIBLE TOLERANCE	SIGMA-R	SIGMA-L	SIGMA-L/SIGMA-R
IRON	83-2	17	17	51	51	66.8144	.1928	.2372	.08814	.08858	1.01
IRON	78-12	17	16	51	48	60.6881	.2065	.2787	.07295	.08388	1.15
IRON	81-2	17	16	51	48	59.6357	.1816	.3119	.08418	.10043	1.56
IRON	77-8	15	15	45	45	45.9275	.2544	.3017	.08988	.08560	.95

THE REGRESSION EQUATIONS ARE:

CORR. COEFF.

R	=	-.0032 X +	.3844								
P	=	-.0025 X +	.4309								
SIGMA-R	=	-.0011 X +	.1393								
SIGMA-L	=	-.0006 X +	.1170								

-.8763

-.6769

-.8762

-.3759

Table 1c

150/TC 102/SC 2/NG 23A - TOTAL IRON BY POLLUTION FREE TICL3 METHOD

SUMMARY OF PRECISIONS FOR ALL SAMPLES

ELEMENT	SAMPLE	KO	K	NO	N	MEAN(X)	REPEATABILITY	PERMISSIBLE TOLERANCE	SIGMA-R	SIGMA-L	SIGMA-L/SIGMA-R
IRON	78-17	34	32	110	105	67.1818	.2198	.5029	.07759	.16902	2.18
IRON	83-2	35	30	113	98	68.7471	.1698	.2944	.06002	.09497	1.58
IRON	78-12	35	34	113	111	60.6684	.2831	.2880	.10004	.06297	.63
IRON	81-2	34	34	110	110	58.5675	.1942	.2983	.06860	.08357	1.36
IRON	77-8	34	29	110	96	45.8620	.1739	.3167	.08145	.10313	1.68

THE REGRESSION EQUATIONS ARE:

CORR. COEFF.

R	=	.0012 X +	.1348								
P	=	.0039 X +	.1019								
SIGMA-R	=	.0004 X +	.0476								
SIGMA-L	=	.0013 X +	.0250								

Table 2a

ISO/TC 102/SC 2/MG 168 - FE-TOTAL TICL3 REDUCTION ACID DISSOLUTION

LIST OF OUTLIERS						
ELEMENT	SAMPLE	COCHRAN (PRECISION)	DIXON (ACCURACY)	LAB HAS ONLY ONE RESULT	INDIVIDUAL OUTLYING RESULT (DIXON)	
IRON	83-2	FR 1	-	-	-	-
		GER 1	-	-	-	-
IRON	76-12	FR 1	-	-	-	-
		GER 1	-	-	-	-
IRON	81-2	FR 1	-	-	-	-
		FR 3	-	-	-	-
		GER 1	-	-	-	-
IRON	77-8	FR 1	-	-	-	-
		JP 1	-	-	-	-

Table 2b

ISO/TC 102/SC 2/HG 178 - FE-TOTAL AG REDUCTION ACID DISSOLUTION

LIST OF OUTLIERS					
ELEMENT	SAMPLE	COCHRAN (PRECISION)	DIXON (ACCURACY)	LAB HAS ONLY ONE RESULT	INDIVIDUAL OUTLYING RESULT (DIXON)
IRON	83-2	-	-	-	-
IRON	78-12	-	SW 3	-	-
IRON	81-2	SW 3	-	-	-
IRON	77-8	-	-	-	-

Table 2c

ISD/TC 102/EC 2/HQ 23A - TOTAL IRON BY POLLUTION FREE TICL3 METHOD

LIST OF OUTLIERS					
ELEMENT	SAMPLE	COCHRAN (PRECISION)	DIXON (ACCURACY)	LAB HAS ONLY ONE RESULT	INDIVIDUAL OUTLYING RESULT (DIXON)
IRON	76-17	BR 3	-	-	-
		GER 3	-	-	-
IRON	83-2	FR 2	SW 1	-	-
		FR 3		-	-
		GER 3		-	-
		SA 1A		-	-
IRON	76-12	BR 3	-	-	-
IRON	81-2	-	-	-	-
IRON	77-8	BR 3	-	-	-
		FR 3	-	-	-
		GER 3	-	-	-
		SA 1A	-	-	-
		SW 1	-	-	-

Table 3a - ISO/TC 102/SC 2/WG 16B. Comparison between means and precision obtained from international tests conducted during the periods of 1980/82 and 1982/84.

Sample	Year of International Test	Grand Mean % 95% Confidence Interval			Precision %			
		Low	Mean	High	r	P	σ_r	σ_L
83-2	1984	66.708	66.758	66.809	0.1522	0.2668	0.0538	0.0863
	1982	66.726	66.757	66.788	0.1668	0.1991	0.0589	0.0567
76-12	1984	60.579	60.630	60.682	0.1657	0.2724	0.0585	0.0869
	1982	60.623	60.674	60.725	0.1945	0.3155	0.0687	0.1003
81-2	1984	59.518	59.560	59.601	0.1213	0.2093	0.0428	0.0674
	1982	59.572	59.606	59.640	0.1635	0.2052	0.0578	0.0599

Table 3b - ISO/TC 102/SC 2/WG 17B. Comparison between means and precision obtained from international tests conducted during the periods of 1980/82 and 1982/84.

Sample	Year of International Test	Grand Mean % 95% Confidence Limits			Precision %			
		Low	Mean	High	r	P	σ_r	σ_L
83-2	1984	66.774	66.814	66.855	0.1928	0.2372	0.0681	0.0686
	1982	66.741	66.780	66.820	0.2214	0.3303	0.0782	0.1028
76-12	1984	60.619	60.669	60.719	0.2065	0.2787	0.0729	0.0839
	1982	60.602	60.648	60.693	0.2149	0.3784	0.0759	0.1224
81-2	1984	59.579	59.636	59.693	0.1816	0.3119	0.0642	0.1004
	1982	59.577	59.623	59.648	0.2119	0.2972	0.0749	0.0907

Table 4a. ISO/TC 102/SC 2/WG 16B , 17B and 23A. Comparison between the grand means, their confidence limits and precision obtained from the data resulting from international tests conducted by WG 16B, WG 17B and WG 23A during the period of 1982/84.

Sample	Working Group	Grand Mean % 95% Confidence Limits			Precision %				σ_L/σ_r
		Low	Mean	High	r	P	σ_r	σ_L	
83-2	WG 16B	66.708	66.758	66.809	0.1522	0.2668	0.0538	0.0863	1.6
	WG 17B	66.774	66.814	66.855	0.1928	0.2372	0.0681	0.0686	1.0
	WG 23A	66.708	66.747	66.786	0.1699	0.2944	0.0600	0.0950	1.6
76-12	WG 16B	60.579	60.630	60.682	0.1657	0.2724	0.0585	0.0869	1.5
	WG 17B	60.619	60.669	60.719	0.2065	0.2787	0.0729	0.0839	1.2
	WG 23A	60.638	60.668	60.698	0.2831	0.2680	0.1000	0.0630	0.6
81-2	WG 16B	59.518	59.560	59.601	0.1213	0.2093	0.0428	0.0674	1.6
	WG 17B	59.579	59.636	59.693	0.1816	0.3119	0.0642	0.1004	1.6
	WG 23A	59.531	59.567	59.604	0.1942	0.2983	0.0686	0.0936	1.4
77-8	WG 16B	45.841	45.880	45.919	0.1849	0.2058	0.0653	0.0562	0.9
	WG 17B	45.872	45.927	45.983	0.2544	0.3017	0.0899	0.0856	1.0
	WG 23A	45.819	45.862	45.905	0.1739	0.3167	0.0614	0.1031	1.7

Table 4b. ISO/TC 102/SC 2/WG 16B, WG 17B, WG 23A. Relative comparison between means and precision of analytical results obtained from international tests conducted during the period of 1982/84. For a given sample, all variables are normalized to WG 16B.

Sample	Working Group	Grand Mean	Precision			
			r	P	σ_x	σ_L
83-2	WG 16B	100.0	100	100	100	100
	WG 17B	100.1	127	89	127	79
	WG 23A	100.0	112	110	111	110
76-12	WG 16B	100.0	100	100	100	100
	WG 17B	100.1	125	102	125	96
	WG 23A	100.1	171	98	171	72
81-2	WG 16B	100.0	100	100	100	100
	WG 17B	100.1	150	149	150	140
	WG 23A	100.0	160	142	160	130
77-8	WG 16B	100.0	100	100	100	100
	WG 17B	100.1	139	147	138	152
	WG 23A	100.0	94	154	94	183

Table 5. Fe-total pollution free titrimetric methods. Average precision of the analytical results obtained from international tests conducted during the period of 1982/84

Precision	TiCl ₃ reduction WG 16B	Ag reduction WG 17B	TiCl ₃ reduction Perchloric Acid Oxidation WG 23A
r%	0.156	0.209	0.203
P%	0.239	0.282	0.294
σ_r %	0.055	0.074	0.073
σ_L %	0.074	0.085	0.089

Table 6. Precision of ISO 2597 estimated from international test data reported in document ISO/TC 102/SC 2 N 147 E⁸.

Reducing Agent	r^* %	P %	σ_r %	σ_L %	σ_L/σ_r
Sn Cl ₂	0.238	0.515	0.084	0.172	2.05
H ₂ S	0.263	0.497	0.093	0.163	1.75

* Conversion factor of 2.77 has been replaced by 2.83.

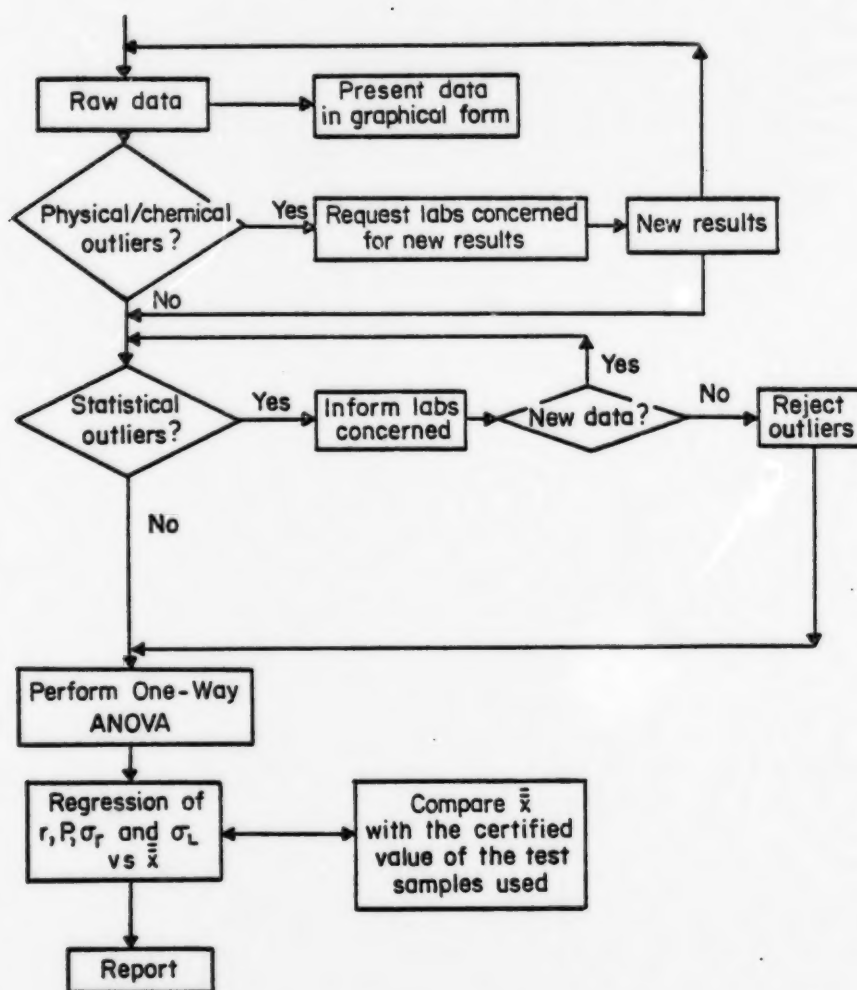


Figure 1. Flow Chart of the Procedure for Statistical Evaluation of Analytical Data Resulting from International Tests If The Test Sample is Known to be Homogeneous.

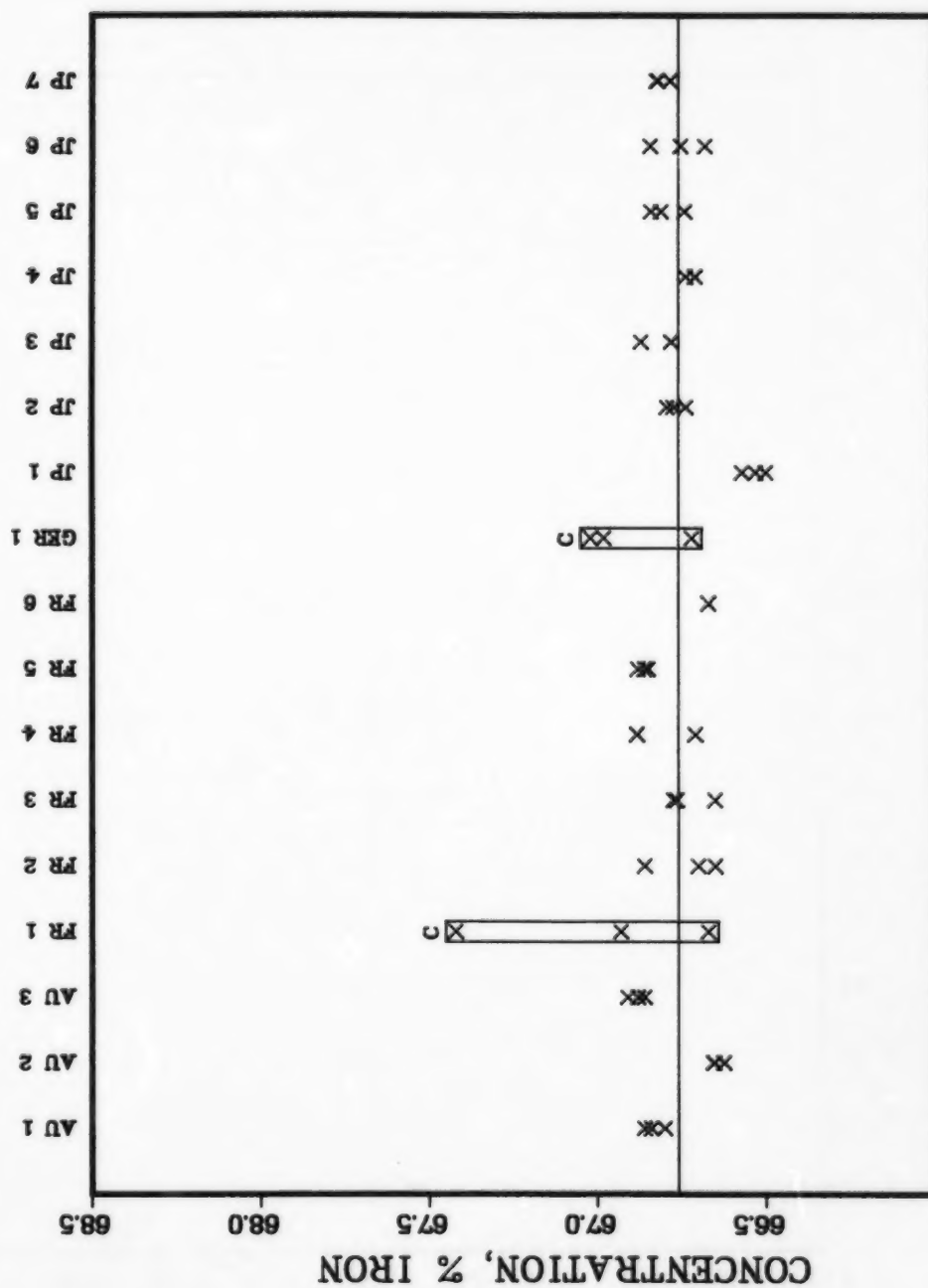


Figure 2.a.1

ISO/TC 102/SC 2/WG 16B - FE-TOTAL TICL3 REDUCTION ACID DISSOLUTION

SAMPLE 83-2 JSS 850-3 IRON WT %

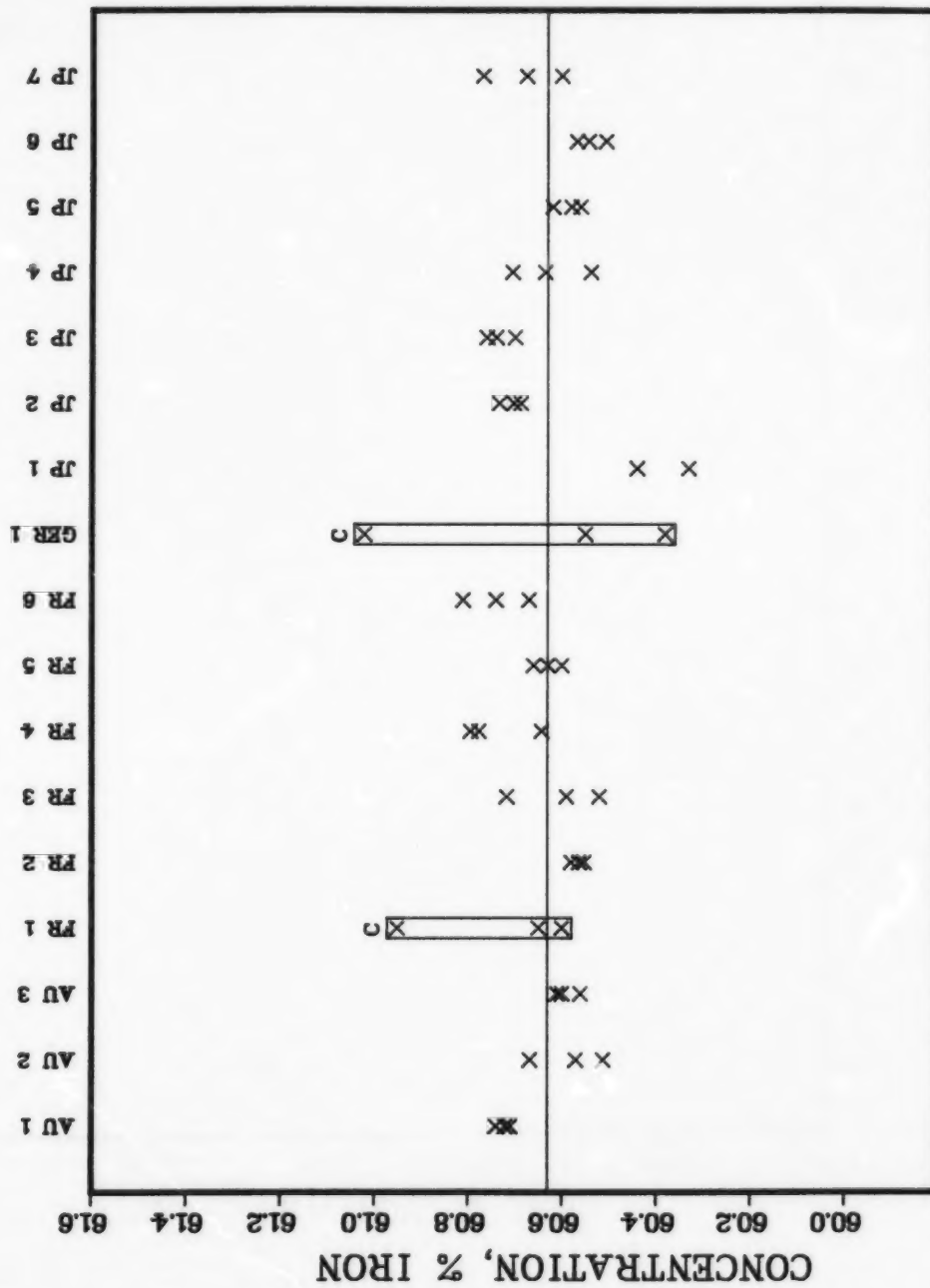


Figure 2.a.2

ISO/TC 102/SC 2/WG 16B - FE-TOTAL TICL3 REDUCTION ACID DISSOLUTION

SAMPLE 76-12 SCH-1 IRON WT %

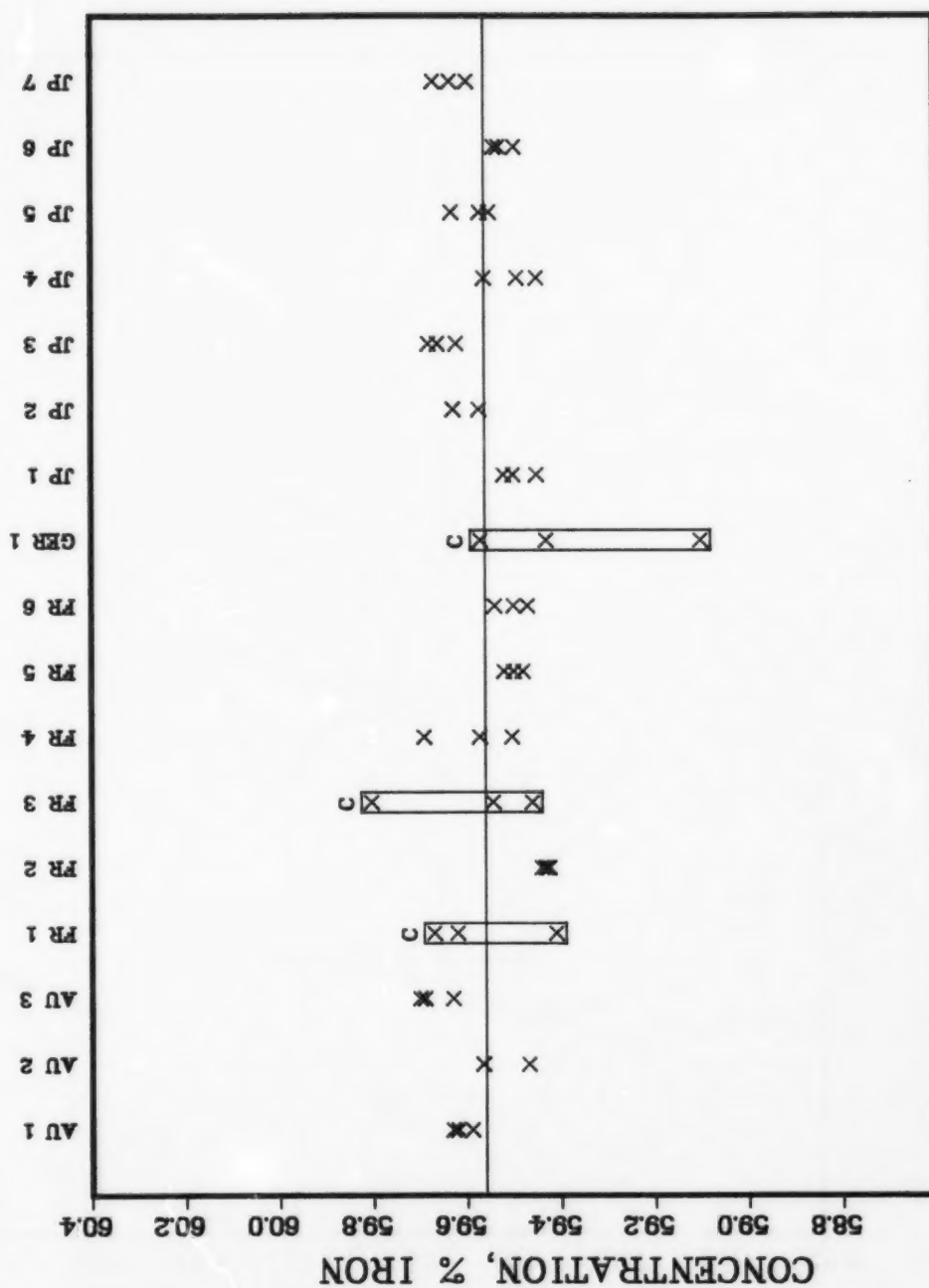


Figure 2.a.3

ISO/TC 102/SC 2/WG 16B - FE-TOTAL TICL3 REDUCTION ACID DISSOLUTION

SAMPLE: 81-2 NBS 692

IRON

WT %

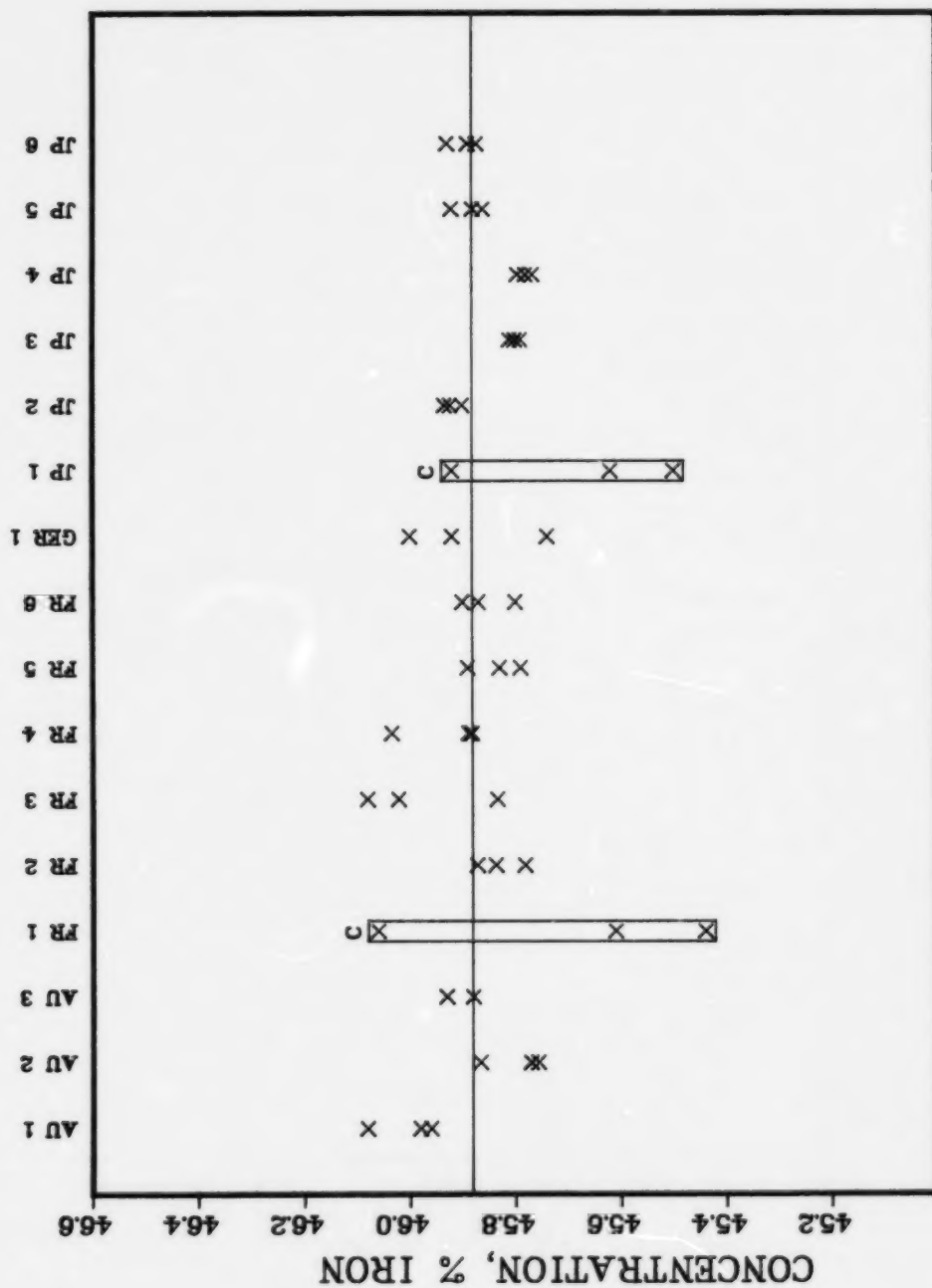


Figure 2.a.4

ISO/TC 102/SC 2/WG 16B - FE-TOTAL TICL3 REDUCTION ACID DISSOLUTION

SAMPLE: 77-8 LM-07 IRON WT %

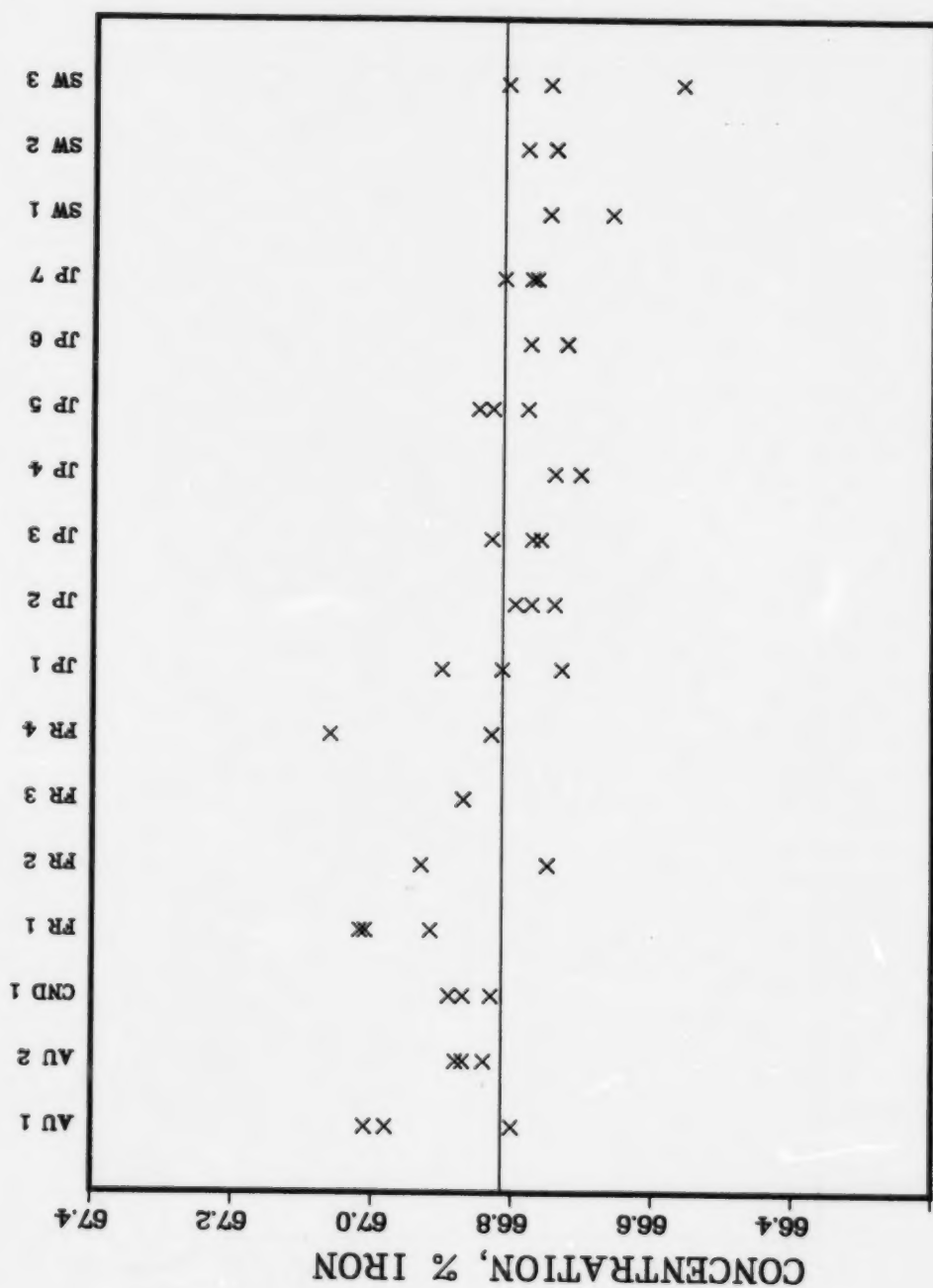


Figure 2.b.1

ISO/TC 102/SC 2/WG 17B - FE-TOTAL AG REDUCTION ACID DISSOLUTION

SAMPLE: 83-2 JSS 850-3 IRON WT %

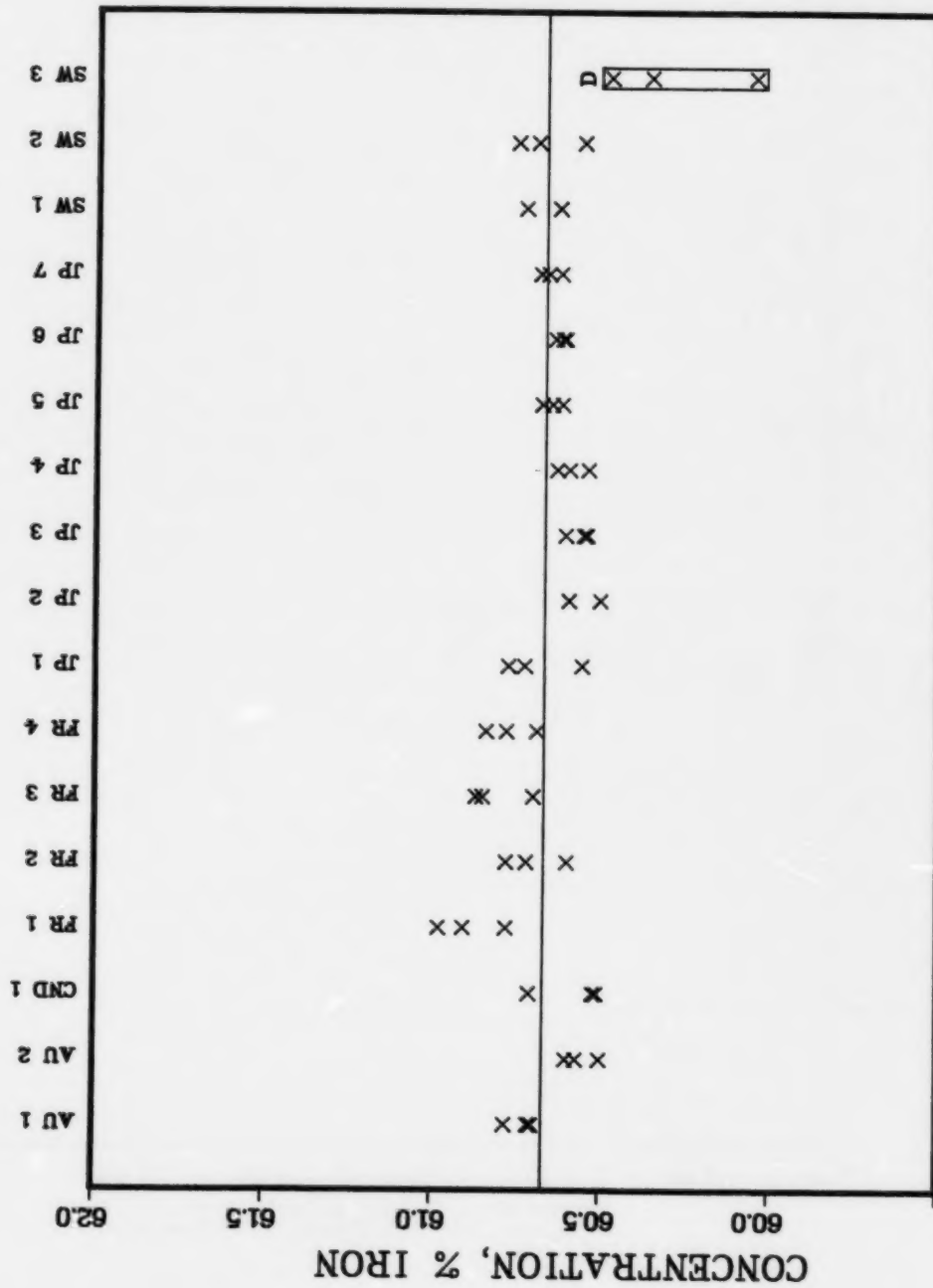


Figure 2.b.2

ISO/TC 102/SC 2/WG 17B - FE-TOTAL AG REDUCTION ACID DISSOLUTION

SAMPLE 78-12 SCH-1

IRON WT %

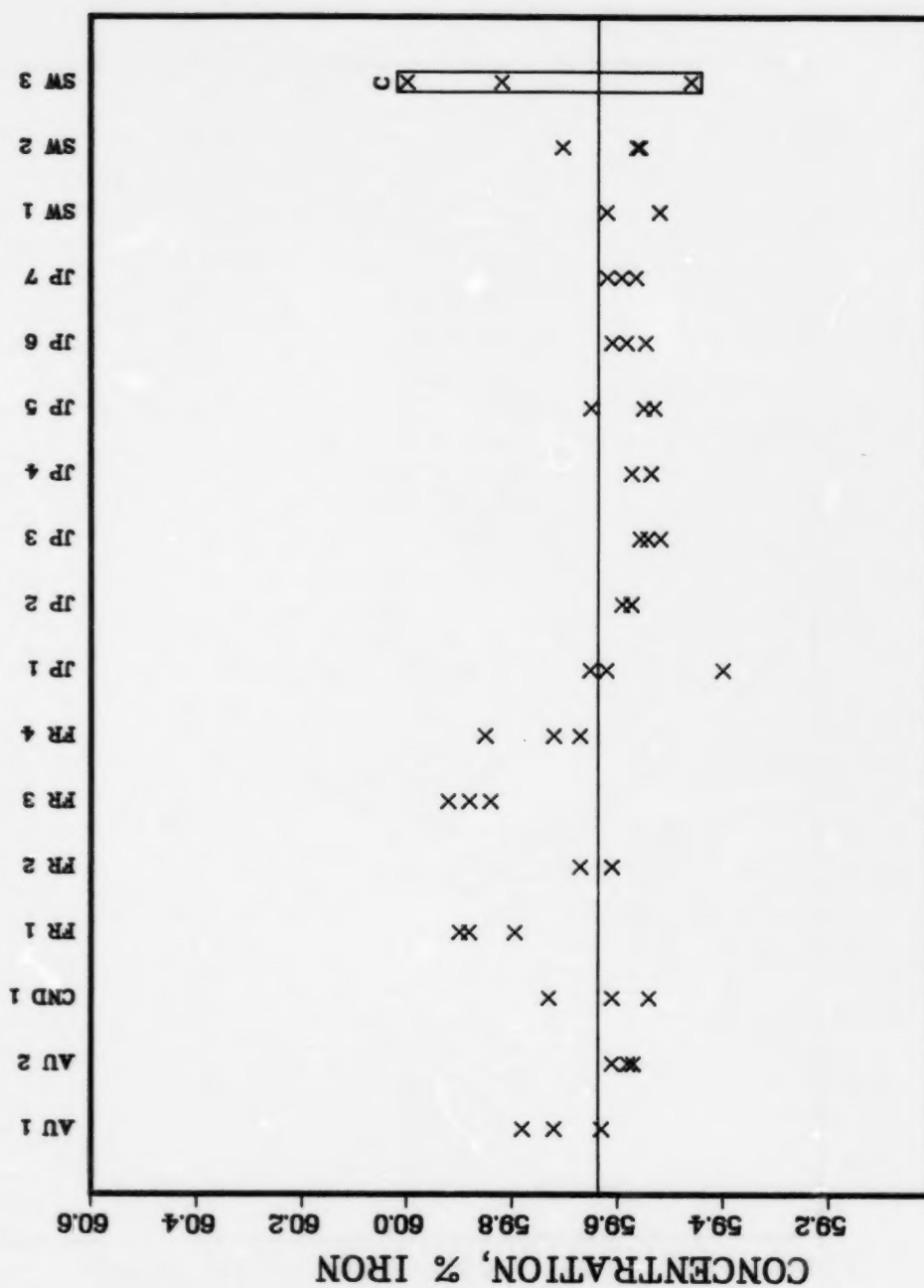


Figure 2.b.b.3

ISO/TC 102/SC 2/WG 17B - FE-TOTAL AG REDUCTION ACID DISSOLUTION

SAMPLE: 81-2 NBS 692 IRON WT %

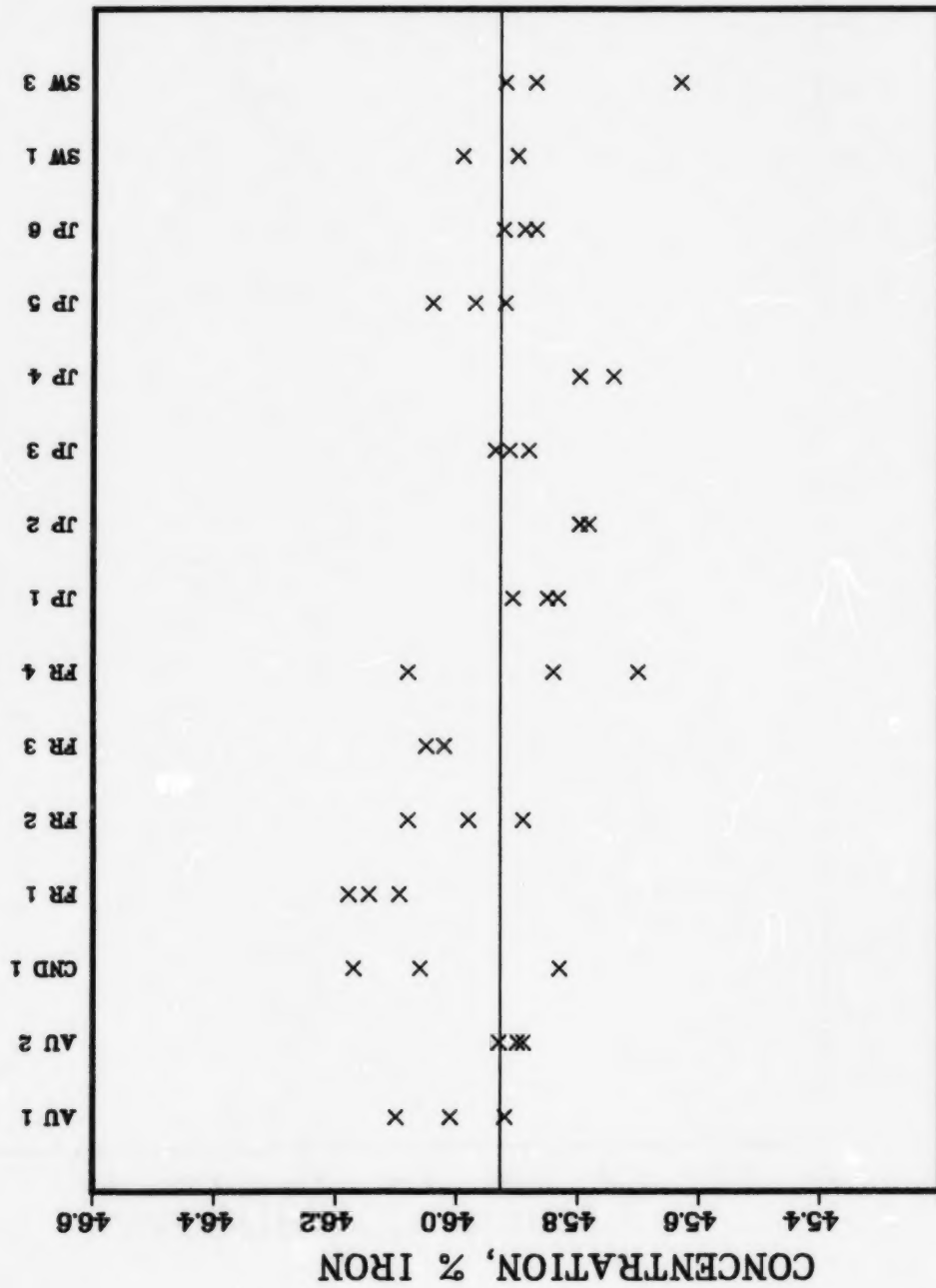


Figure 2.b.4

ISO/TC 102/SC 2/WG 17B - FE-TOTAL AG REDUCTION ACID DISSOLUTION

SAMPLE: 77-8 LM-07

IRON WT %

AU 1
 AU 2
 AU 3
 AU 4
 BR 1
 BR 2
 BR 3
 CND 1
 CND 2
 CND 3
 FR 1
 FR 2
 FR 3
 FR 4
 FR 5
 FR 6
 GER 1
 GER 2
 GER 3
 IT 1
 IT 2
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 SA 1A
 SA 2
 SW 1
 USA 1
 USA 2
 USA 3
 USA 4
 USA 5

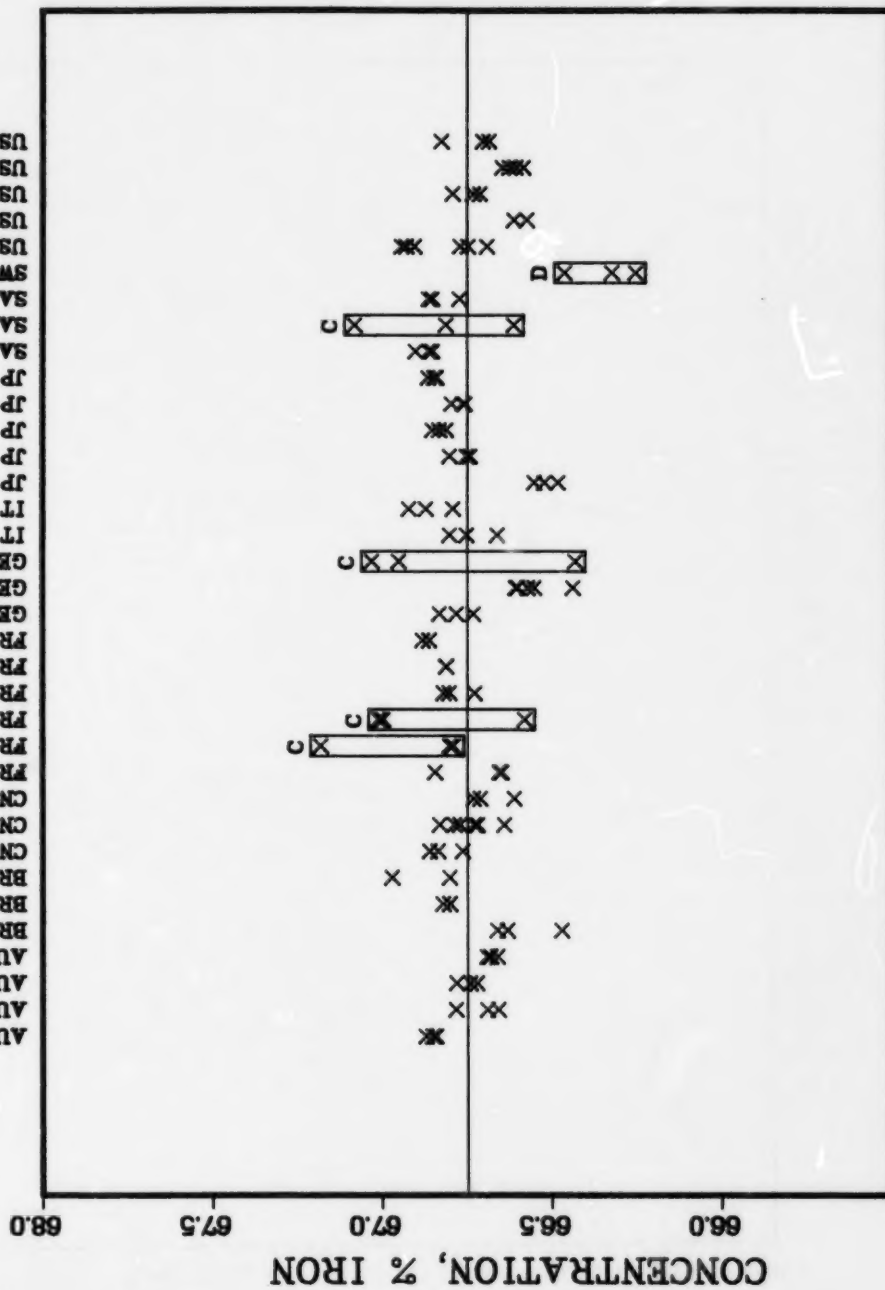
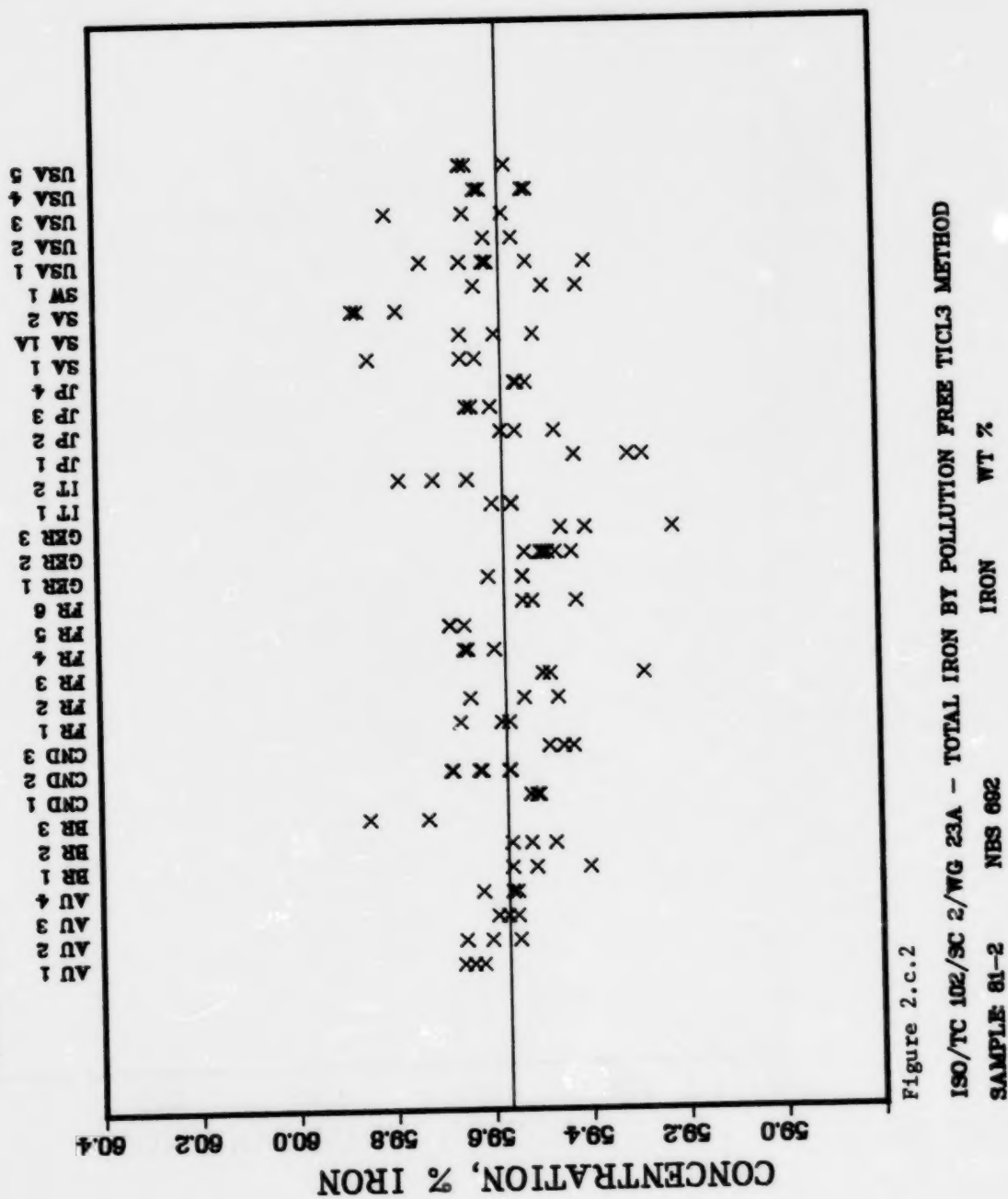


Figure 2.c.1

ISO/TC 102/SC 2/WG 23A - TOTAL IRON BY POLLUTION FREE TICLS METHOD

SAMPLE 83-2 JSS 850-3

IRON WT %



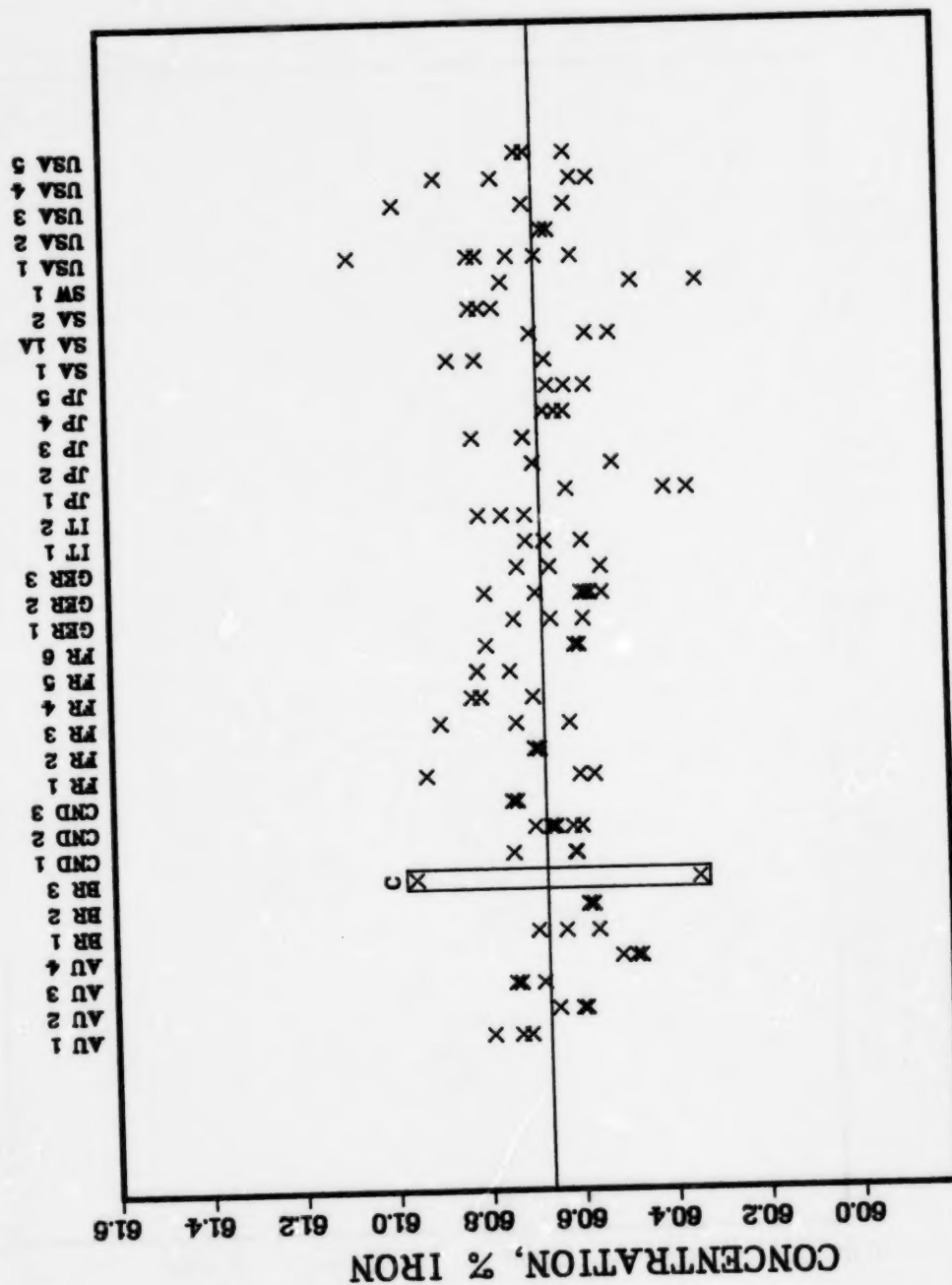


Figure 2.c.3
ISO/TC 102/SC 2/WG 23A - TOTAL IRON BY POLLUTION FREE TICL3 METHOD
SAMPLE 76-12 SCH-1 IRON WT %

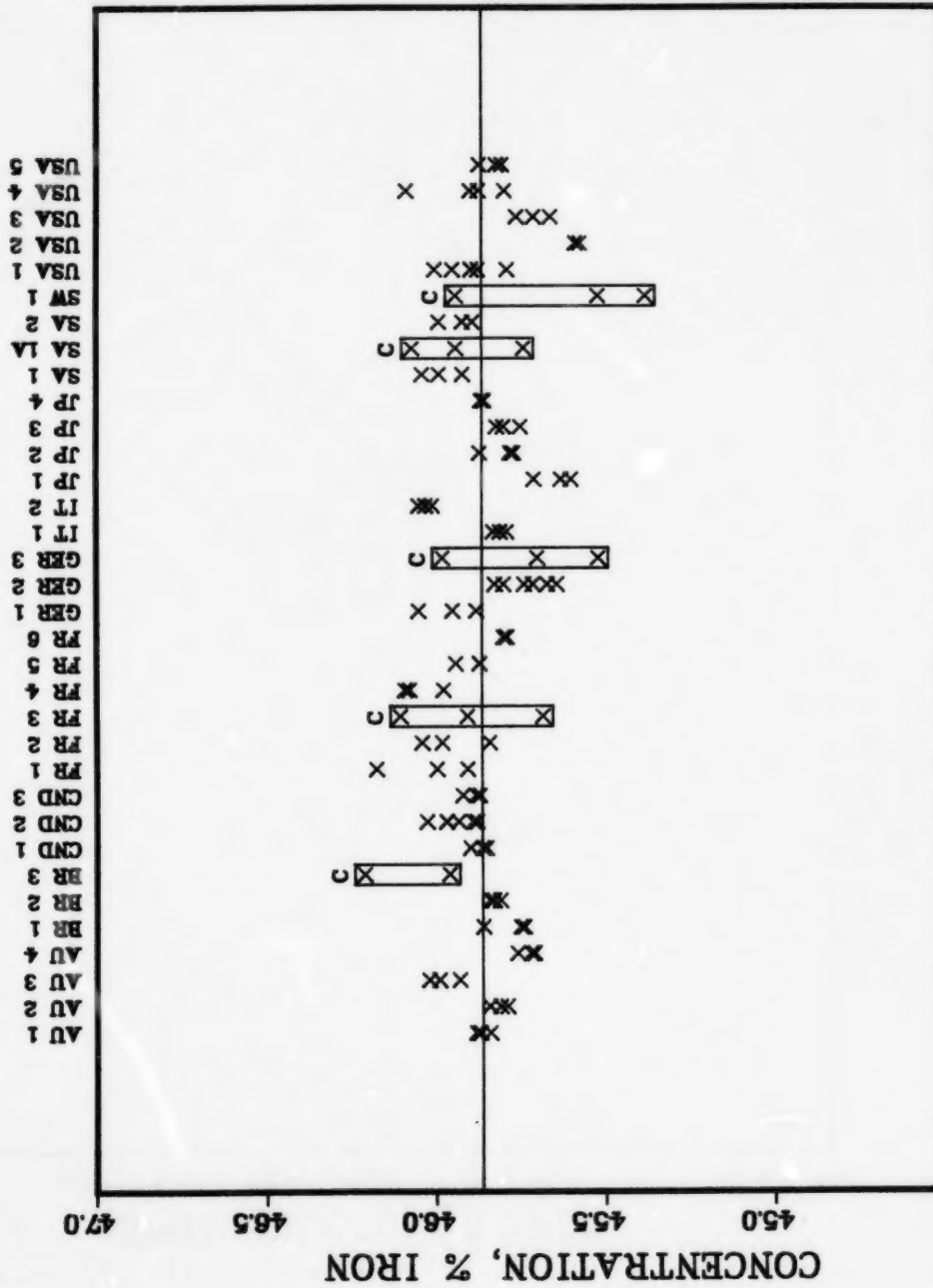


Figure 2.c.4

ISO/TC 102/SC 2/WG 23A - TOTAL IRON BY POLLUTION FREE TICL3 METHOD

SAMPLE: 77-8 LM-07 IRON WT %

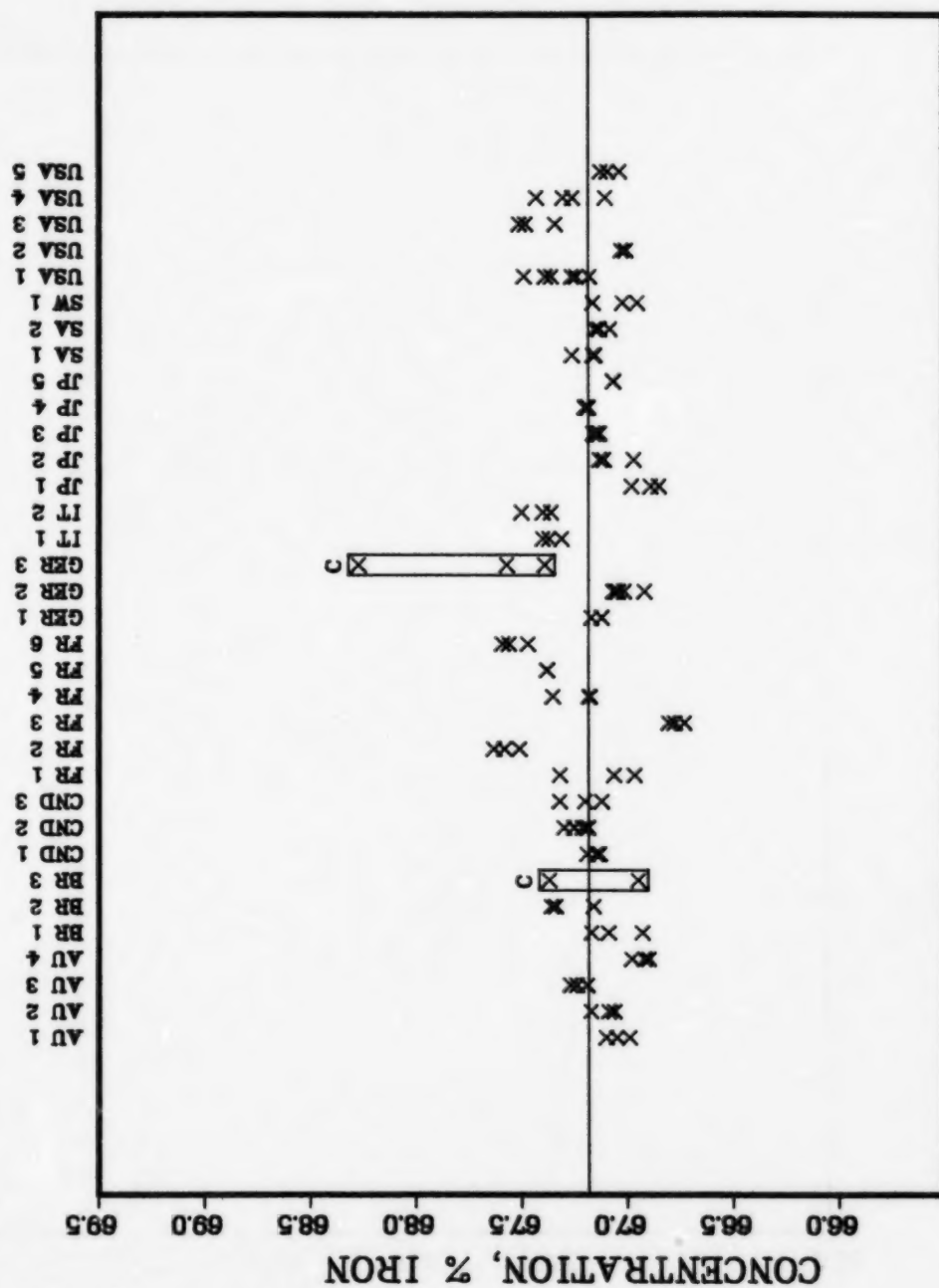


Figure 2.c.5

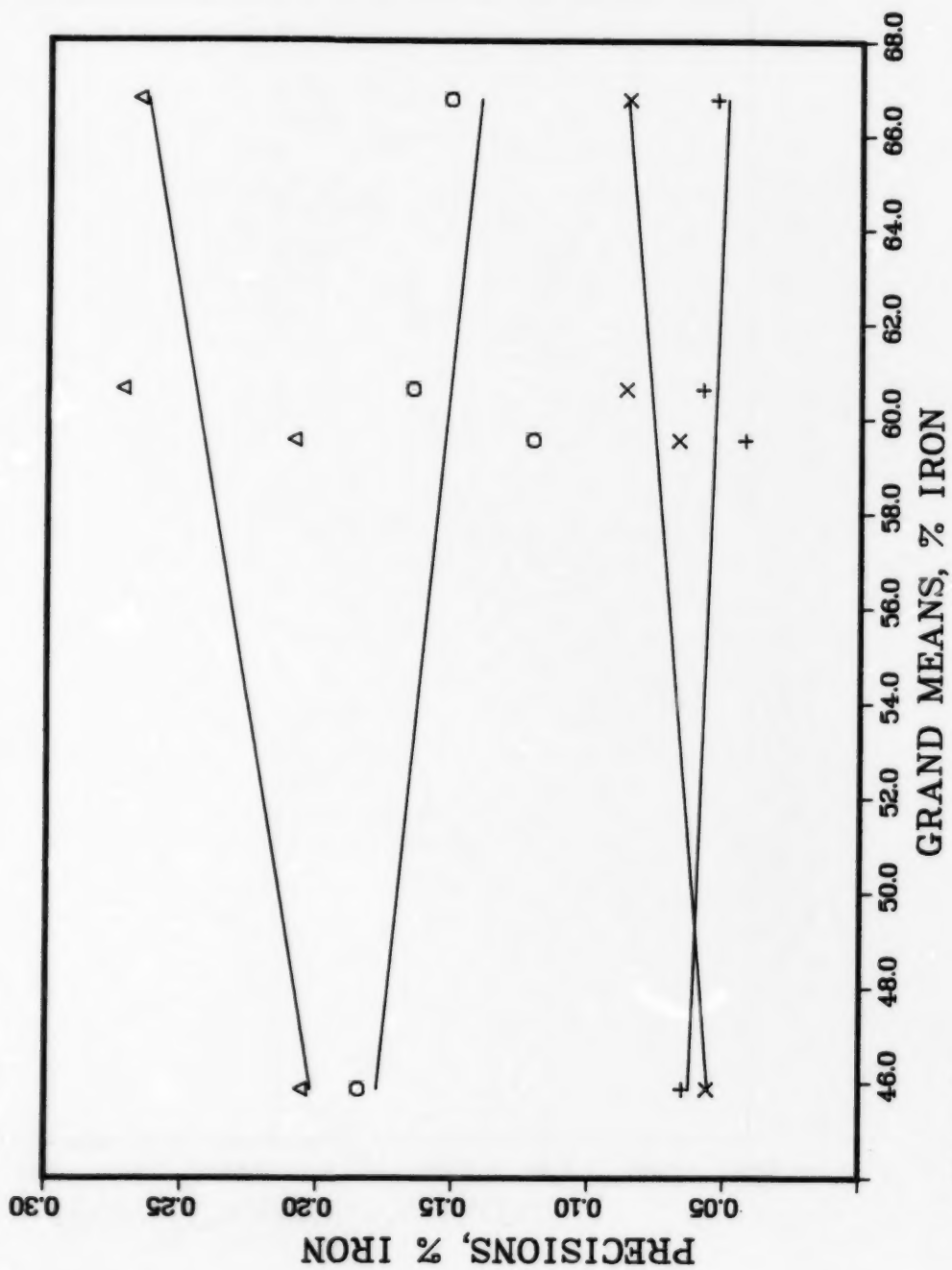


Figure 3.a

ISO/TC 102/SC 2/WG 16B - FE-TOTAL TiCl3 REDUCTION ACID DISSOLUTION

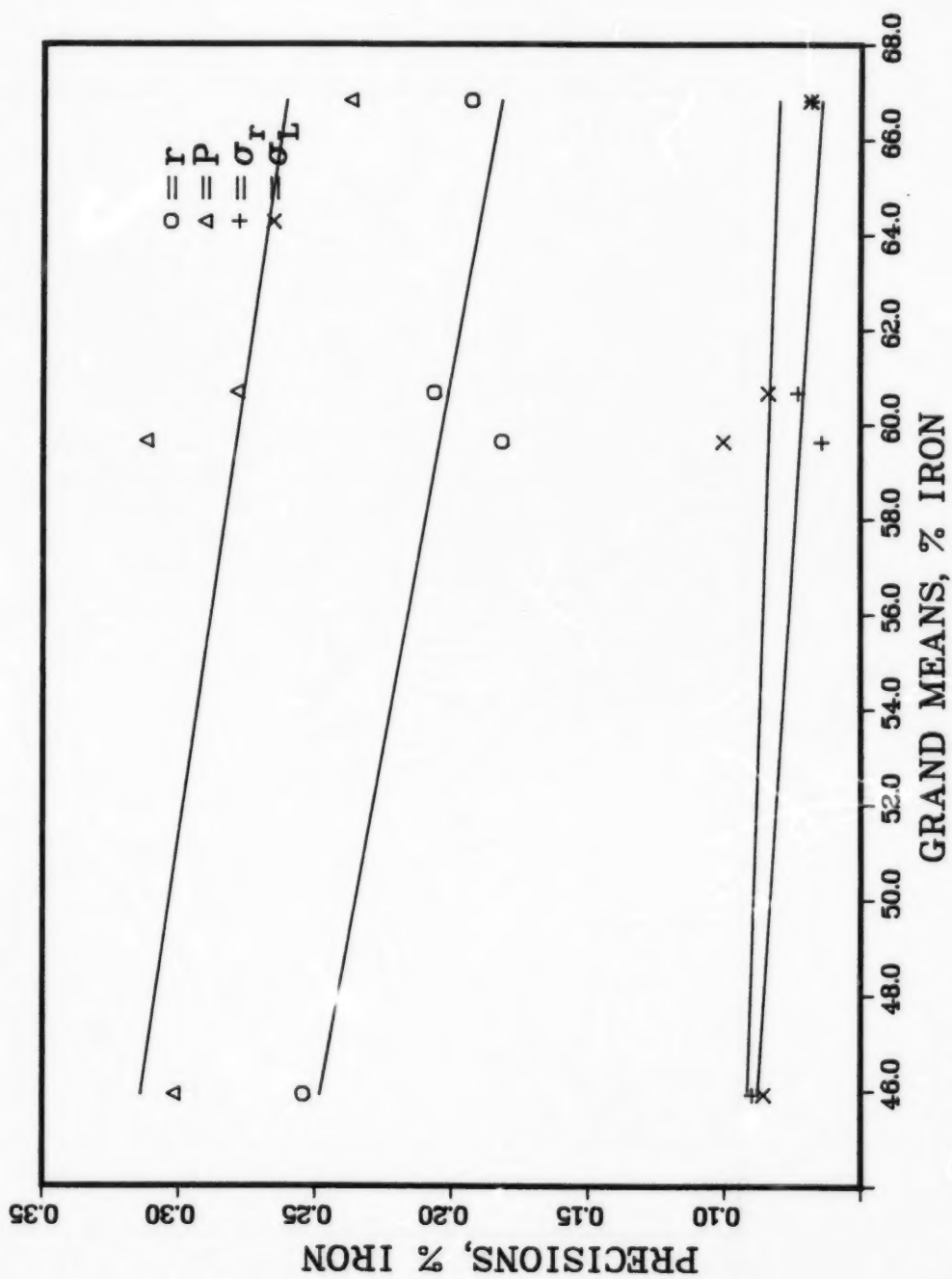


Figure 3.b

ISO/TC 102/SC 2/WG 17B - FE-TOTAL AG REDUCTION ACID DISSOLUTION

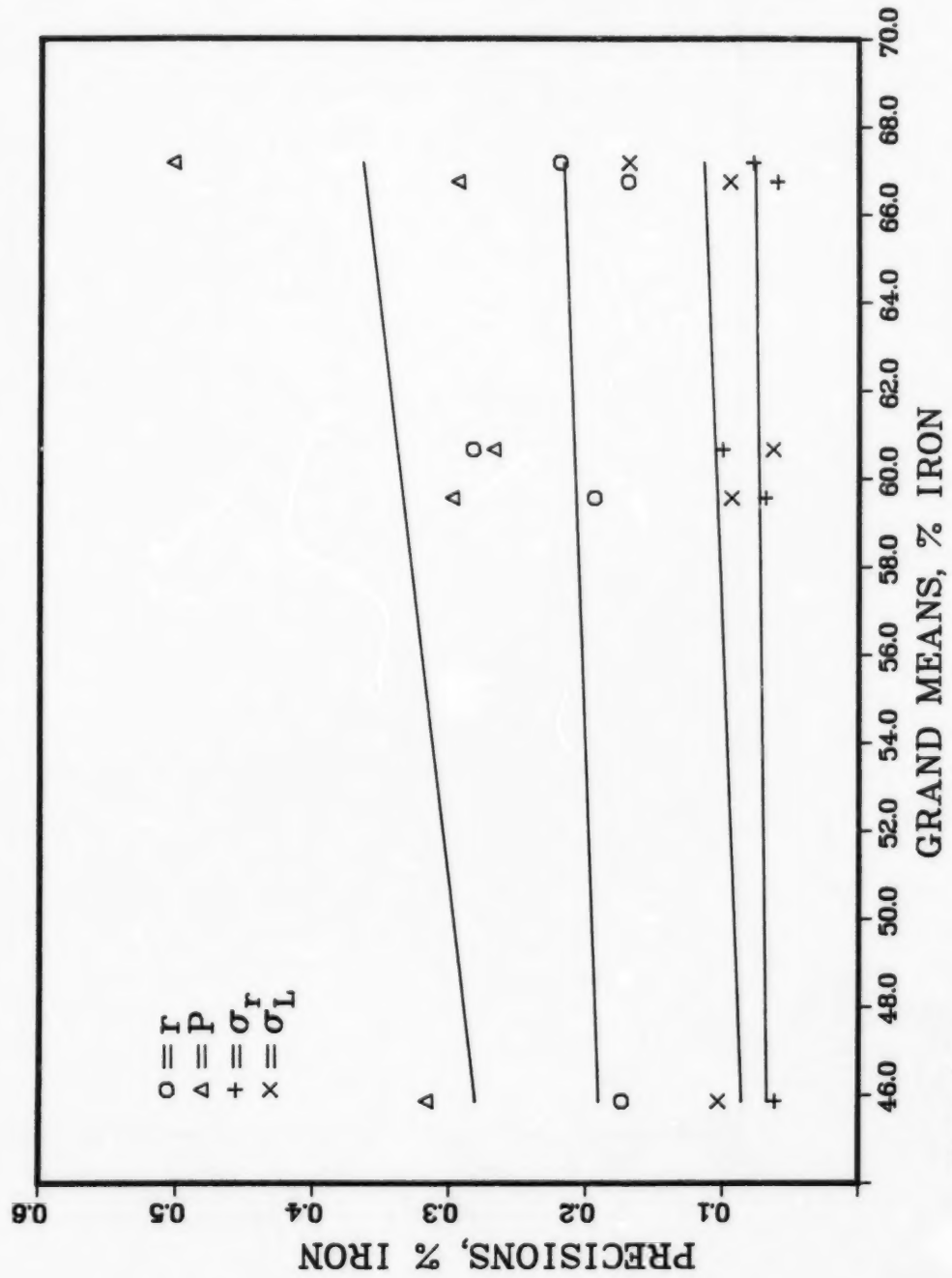


Figure 3.c

ISO/TC 102/SC 2/WG 23A - TOTAL IRON BY POLLUTION FREE TICL3 METHOD

ISO/TC 102/SC 2 WG 16B

Fe-total - TiCl_3 Reduction - Acid Dissolution

APPENDIX A1

DETAILED COMPUTATION OF ANALYTICAL DATA

A1-1

ISO/TC 102/SC 2/WG 16B - FE-TOTAL TICL3 REDUCTION ACID DISSOLUTION
SAMPLE: 83-2

LABORATORY			BOTTLE			ANALYTICAL RESULTS			LAB	LAB
NO.	COUNTRY	NO.							MEAN	VARIANCE
1	AU	1	1	66.8600	66.8400	66.8000			66.83333	.9333E-03
2	AU	2	1	66.6560	66.6530	66.6240			66.64433	.3123E-03
3	AU	3	1	66.8600	66.9100	66.8800			66.88333	.6333E-03
4	FR	1	1	67.4200	66.6700	66.9300			67.00667	.1450E+00
5	FR	2	1	66.6500	66.7000	66.8590			66.73633	.1191E-01
6	FR	3	1	66.7740	66.7640	66.6510			66.72967	.4666E-02
7	FR	4	1	66.7088	66.8814	66.8826			66.82427	.1000E-01
8	FR	5	1	66.8500	66.8800	66.8600			66.86333	.2333E-03
9	FR	6	1	66.6700	66.6700	66.6700			66.67000	0.
10	GER	1	1	67.0200	66.9800	66.7200			66.90667	.2653E-01
11	JP	1	1	66.5300	66.5000	66.5700			66.53333	.1233E-02
12	JP	2	1	66.7370	66.7930	66.7740			66.76800	.8110E-03
13	JP	3	1	66.8700	66.7800	66.7800			66.81000	.2700E-02
14	JP	4	1	66.7370	66.7090	66.7090			66.71833	.2613E-03
15	JP	5	1	66.7400	66.8400	66.8100			66.79667	.2633E-02
16	JP	6	1	66.8420	66.7520	66.6800			66.75800	.6588E-02
17	JP	7	1	66.8190	66.8230	66.7830			66.80833	.4853E-03

ISO/TC 102/SC 2/WG 16B - FE-TOTAL TICL3 REDUCTION ACID DISSOLUTION
SAMPLE: 83-2

BARTLETT'S TEST FOR HOMOGENEITY OF LABORATORY VARIANCE

CALCULATED STATISTIC	48.176
CHI-SQUARE(.95,15)	25.000

VARIANCE IS INHOMOGENEOUS

COCHRAN'S TEST FOR LABORATORY VARIANCE

LAB- 4	C-CALC = .675	C-CRIT(3,16) = .319
LAB-10	C-CALC = .379	C-CRIT(3,15) = .334

DIXON'S TEST FOR LABORATORY MEANS

NONE REJECTED

LIST OF LABORATORIES WHOSE RESULTS WERE NOT USED FOR ONE-WAY ANOVA

LAB- 4	COCHRAN'S TEST FOR LABORATORY VARIANCE
LAB-10	COCHRAN'S TEST FOR LABORATORY VARIANCE

LIST OF INDIVIDUAL OUTLIERS DETECTED BY DIXON'S TEST WITHIN-LABORATORIES

NONE REJECTED

Al-3

ISO/TC 102/SC 2/WG 16B - FE-TOTAL TICL3 REDUCTION ACID DISSOLUTION
SAMPLE: 83-2

ONE-WAY ANALYSIS OF VARIANCE TABLE

<u>SOURCE OF VARIATION</u>	<u>D.F.</u>	<u>SUM OF SQUARES</u>	<u>MEAN SQUARE</u>
BETWEEN LABS	14	.35305159E+00	.25217971E-01
WITHIN LABS	30	.86802213E-01	.28934071E-02
TOTAL	44	.43985380E+00	

REPEATABILITY VARIANCE	.28934071E-02
REPEATABILITY STD. DEV.	.05379040

BETWEEN-LABORATORIES VARIANCE	.74415212E-02
BETWEEN-LABORATORIES STD. DEV.	.08626425

REPEATABILITY	.15222683
PERMISSIBLE TOLERANCE BETWEEN LABS	.26680499

GRAND MEAN	66.75848444
VARIANCE OF THE GRAND MEAN	.56039935E-03

ISO/TC 102/SC 2/WG 16B - FE-TOTAL TICL3 REDUCTION ACID DISSOLUTION
SAMPLE: 76-12

LABORATORY			BOTTLE			ANALYTICAL RESULTS			LAB	LAB
NO.	COUNTRY	NO.							MEAN	VARIANCE
1	AU	1	1	60.7400	60.7200	60.7100			60.72333	.2333E-03
2	AU	2	1	60.6680	60.5700	60.5120			60.58333	.6217E-02
3	AU	3	1	60.6000	60.6100	60.5600			60.59000	.7000E-03
4	FR	1	1	60.9500	60.6000	60.6500			60.73333	.3583E-01
5	FR	2	1	60.5520	60.5630	60.5800			60.56500	.1990E-03
6	FR	3	1	60.7170	60.5900	60.5200			60.60900	.9973E-02
7	FR	4	1	60.6433	60.7939	60.7755			60.73757	.6749E-02
8	FR	5	1	60.6600	60.6000	60.6300			60.63000	.9000E-03
9	FR	6	1	60.8100	60.6700	60.7400			60.74000	.4900E-02
10	GER	1	1	61.0200	60.5500	60.3800			60.65000	.1099E+00
11	JP	1	1	60.4400	60.3300	60.4400			60.40333	.4033E-02
12	JP	2	1	60.7340	60.6880	60.7030			60.70833	.5503E-03
13	JP	3	1	60.7600	60.7400	60.7000			60.73333	.9333E-03
14	JP	4	1	60.7060	60.6360	60.5380			60.62667	.7121E-02
15	JP	5	1	60.5600	60.5800	60.6200			60.58667	.9333E-03
16	JP	6	1	60.5430	60.5680	60.5060			60.53900	.9730E-03
17	JP	7	1	60.7670	60.6750	60.6000			60.68067	.6996E-02

ISO/TC 102/SC 2/WG 16B - FE-TOTAL TICL3 REDUCTION ACID DISSOLUTION
SAMPLE: 76-12

BARTLETT'S TEST FOR HOMOGENEITY OF LABORATORY VARIANCE

CALCULATED STATISTIC	42.074
CHI-SQUARE(.95,16)	26.301

VARIANCE IS INHOMOGENEOUS

COCHRAN'S TEST FOR LABORATORY VARIANCE

LAB-10	C-CALC = .557	C-CRIT(3,17) = .305
LAB- 4	C-CALC = .411	C-CRIT(3,16) = .319

DIXON'S TEST FOR LABORATORY MEANS

NONE REJECTED

LIST OF LABORATORIES WHOSE RESULTS WERE NOT USED FOR ONE-WAY ANOVA

LAB- 4	COCHRAN'S TEST FOR LABORATORY VARIANCE
LAB-10	COCHRAN'S TEST FOR LABORATORY VARIANCE

LIST OF INDIVIDUAL OUTLIERS DETECTED BY DIXON'S TEST WITHIN-LABORATORIES

NONE REJECTED

ISO/TC 102/SC 2/WG 16B - FE-TOTAL TICL3 REDUCTION ACID DISSOLUTION
 SAMPLE: 76-12

ONE-WAY ANALYSIS OF VARIANCE TABLE

<u>SOURCE OF VARIATION</u>	<u>D.F.</u>	<u>SUM OF SQUARES</u>	<u>MEAN SQUARE</u>
BETWEEN LABS	14	.36525466E+00	.26089619E-01
WITHIN LABS	30	.10282592E+00	.34275307E-02
TOTAL	44	.46808058E+00	

REPEATABILITY VARIANCE
 REPEATABILITY STD. DEV.

.34275307E-02
 .05854512

BETWEEN-LABORATORIES VARIANCE
 BETWEEN-LABORATORIES STD. DEV.

.75540293E-02
 .08691392

REPEATABILITY
 PERMISSIBLE TOLERANCE BETWEEN LABS

.16568268
 .27244236

GRAND MEAN
 VARIANCE OF THE GRAND MEAN

60.63041556
 .57976930E-03

ISO/TC 102/SC 2/WG 168 - FE-TOTAL TICL3 REDUCTION ACID DISSOLUTION
 SAMPLE: 81-2

LABORATORY			BOTTLE			ANALYTICAL RESULTS			LAB	LAB
NO.	COUNTRY	NO.							MEAN	VARIANCE
1	AU	1	1	59.6200	59.6300	59.5900			59.61333	.4333E-03
2	AU	2	1	59.5660	59.5660	59.4690			59.53367	.3136E-02
3	AU	3	1	59.7000	59.6900	59.6300			59.67333	.1433E-02
4	FR	1	1	59.6200	59.6700	59.4100			59.56667	.1903E-01
5	FR	2	1	59.4410	59.4250	59.4320			59.43267	.6433E-04
6	FR	3	1	59.8030	59.4610	59.5440			59.60267	.3182E-01
7	FR	4	1	59.5036	59.5720	59.6910			59.58887	.8993E-02
8	FR	5	1	59.4800	59.5000	59.5200			59.50000	.4000E-03
9	FR	6	1	59.5000	59.4700	59.5400			59.50333	.1233E-02
10	GER	1	1	59.4300	59.5700	59.1000			59.36667	.5823E-01
11	JP	1	1	59.5200	59.5000	59.4500			59.49000	.1300E-02
12	JP	2	1	59.6270	59.6270	59.5720			59.60867	.1008E-02
13	JP	3	1	59.6800	59.6600	59.6200			59.65333	.9333E-03
14	JP	4	1	59.4490	59.5610	59.4910			59.50033	.3201E-02
15	JP	5	1	59.5500	59.5700	59.6300			59.58333	.1733E-02
16	JP	6	1	59.5300	59.5400	59.4960			59.52200	.5320E-03
17	JP	7	1	59.6330	59.5970	59.6690			59.63300	.1296E-02

ISO/TC 102/SC 2/WG 168 - FE-TOTAL TICL3 REDUCTION ACID DISSOLUTION
SAMPLE: 81-2

BARTLETT'S TEST FOR HOMOGENEITY OF LABORATORY VARIANCE

CALCULATED STATISTIC	40.411
CHI-SQUARE(.95,16)	26.301

VARIANCE IS INHOMOGENEOUS

COCHRAN'S TEST FOR LABORATORY VARIANCE

LAB-10	C-CALC = .432	C-CRIT(3,17) = .305
LAB- 6	C-CALC = .416	C-CRIT(3,16) = .319
LAB- 4	C-CALC = .426	C-CRIT(3,15) = .334

DIXON'S TEST FOR LABORATORY MEANS

NONE REJECTED

LIST OF LABORATORIES WHOSE RESULTS WERE NOT USED FOR ONE-WAY ANOVA

LAB- 4	COCHRAN'S TEST FOR LABORATORY VARIANCE
LAB- 6	COCHRAN'S TEST FOR LABORATORY VARIANCE
LAB-10	COCHRAN'S TEST FOR LABORATORY VARIANCE

LIST OF INDIVIDUAL OUTLIERS DETECTED BY DIXON'S TEST WITHIN-LABORATORIES

NONE REJECTED

ISO/TC 102/SC 2/WG 16B - FE-TOTAL TICL3 REDUCTION ACID DISSOLUTION
 SAMPLE: 81-2

ONE-WAY ANALYSIS OF VARIANCE TABLE

SOURCE OF VARIATION	D.F.	SUM OF SQUARES	MEAN SQUARE
BETWEEN LABS	13	.20128919E+00	.15483784E-01
WITHIN LABS	28	.51396107E-01	.18355753E-02
TOTAL	41	.25268530E+00	

REPEATABILITY VARIANCE .18355753E-02
 REPEATABILITY STD. DEV. .04284361

BETWEEN-LABORATORIES VARIANCE .45494030E-02
 BETWEEN-LABORATORIES STD. DEV. .06744926

REPEATABILITY .12124743
 PERMISSIBLE TOLERANCE BETWEEN LABS .20925148

GRAND MEAN 59.55970476
 VARIANCE OF THE GRAND MEAN .36866153E-03

ISO/TC 102/SC 2/WG 16B - FE-TOTAL TICL3 REDUCTION ACID DISSOLUTION
SAMPLE: 77-8

LABORATORY			BOTTLE			ANALYTICAL RESULTS			LAB	LAB
NO.	COUNTRY	NO.							MEAN	VARIANCE
1	AU	1	1	46.0800	45.9600	45.9800			46.00667	.4133E-02
2	AU	2	1	45.8660	45.7710	45.7570			45.79800	.3517E-02
3	AU	3	1	45.9300	45.9300	45.8800			45.91333	.8333E-03
4	FR	1	1	45.6100	46.0600	45.4400			45.70333	.1026E+00
5	FR	2	1	45.8720	45.7820	45.8370			45.83033	.2058E-02
6	FR	3	1	46.0800	46.0210	45.8340			45.97833	.1649E-01
7	FR	4	1	45.8876	45.8811	46.0337			45.93413	.7446E-02
8	FR	5	1	45.7900	45.8300	45.8900			45.83667	.2533E-02
9	FR	6	1	45.8000	45.9000	45.8700			45.85667	.2633E-02
10	GER	1	1	46.0000	45.7400	45.9200			45.88667	.1773E-01
11	JP	1	1	45.5000	45.9200	45.6200			45.68000	.4680E-01
12	JP	2	1	45.9340	45.9230	45.9000			45.91900	.3010E-03
13	JP	3	1	45.8100	45.8000	45.7900			45.80000	.1000E-03
14	JP	4	1	45.7800	45.7950	45.7670			45.78067	.1963E-03
15	JP	5	1	45.9200	45.8600	45.8800			45.88667	.9333E-03
16	JP	6	1	45.9280	45.8890	45.8720			45.89633	.8243E-03

ISO/TC 102/SC 2/WG 16B - FE-TOTAL TICL3 REDUCTION ACID DISSOLUTION
SAMPLE: 77-8

BARTLETT'S TEST FOR HOMOGENEITY OF LABORATORY VARIANCE

CALCULATED STATISTIC	42.894
CHI-SQUARE(.95,15)	25.000

VARIANCE IS INHOMOGENEOUS

COCHRAN'S TEST FOR LABORATORY VARIANCE

LAB- 4	C-CALC = .491	C-CRIT(3,16) = .319
LAB-11	C-CALC = .439	C-CRIT(3,15) = .334

DIXON'S TEST FOR LABORATORY MEANS

NONE REJECTED

LIST OF LABORATORIES WHOSE RESULTS WERE NOT USED FOR ONE-WAY ANOVA

LAB- 4	COCHRAN'S TEST FOR LABORATORY VARIANCE
LAB-11	COCHRAN'S TEST FOR LABORATORY VARIANCE

LIST OF INDIVIDUAL OUTLIERS DETECTED BY DIXON'S TEST WITHIN-LABORATORIES

NONE REJECTED

ISO/TC 102/SC 2/WG 168 - FE-TOTAL TICL3 REDUCTION ACID DISSOLUTION
SAMPLE: 77-8

ONE-WAY ANALYSIS OF VARIANCE TABLE

<u>SOURCE OF VARIATION</u>	<u>D.F.</u>	<u>SUM OF SQUARES</u>	<u>MEAN SQUARE</u>
BETWEEN LABS	13	.17853401E+00	.13733386E-01
WITHIN LABS	28	.11947407E+00	.42669312E-02
TOTAL	41	.29800809E+00	

REPEATABILITY VARIANCE	.42669312E-02
REPEATABILITY STD. DEV.	.06532175

BETWEEN-LABORATORIES VARIANCE	.31554848E-02
BETWEEN-LABORATORIES STD. DEV.	.05617370

REPEATABILITY	.18486056
PERMISSIBLE TOLERANCE BETWEEN LABS	.20581223

GRAND MEAN	45.88024762
VARIANCE OF THE GRAND MEAN	.32698537E-03

ISO/TC 102/SC 2/WG 17B

Fe-total - Silver reduction method - Acid decomposition

APPENDIX A2

DETAILED COMPUTATION OF ANALYTICAL DATA

ISO/TC 102/SC 2/WG 17B - FE-TOTAL AG REDUCTION ACID DISSOLUTION
SAMPLE: 83-2

LABORATORY			BOTTLE			ANALYTICAL RESULTS			LAB	LAB
NO.	COUNTRY	NO.							MEAN	VARIANCE
1	AU 1	1	66.8000	66.9800	67.0100				66.93000	.1290E-01
2	AU 2	1	66.8700	66.8400	66.8800				66.86333	.4333E-03
3	CND 1	1	66.8300	66.8700	66.8900				66.86333	.9333E-03
4	FR 1	1	67.0175	66.9166	67.0097				66.98127	.3152E-02
5	FR 2	1	66.7500	66.7500	66.9300				66.81000	.1080E-01
6	FR 3	1	66.8700	66.8700	66.8700				66.87000	0.
7	FR 4	1	66.8300	66.8300	67.0600				66.90667	.1763E-01
8	JP 1	1	66.7300	66.9010	66.8150				66.81533	.7310E-02
9	JP 2	1	66.7410	66.7740	66.7970				66.77067	.7923E-03
10	JP 3	1	66.7730	66.7610	66.8310				66.78833	.1401E-02
11	JP 4	1	66.7410	66.7040	66.7040				66.71633	.4563E-03
12	JP 5	1	66.8300	66.8500	66.7800				66.82000	.1300E-02
13	JP 6	1	66.7250	66.7240	66.7770				66.74200	.9190E-03
14	JP 7	1	66.8140	66.7680	66.7750				66.78567	.6143E-03
15	SW 1	1	66.7500	66.6600	66.7500				66.72000	.2700E-02
16	SW 2	1	66.7410	66.7820	66.7420				66.75500	.5470E-03
17	SW 3	1	66.8100	66.5600	66.7500				66.70667	.1703E-01

ISO/TC 102/SC 2/WG 17B - FE-TOTAL AG REDUCTION ACID DISSOLUTION
SAMPLE: 83-2

BARTLETT'S TEST FOR HOMOGENEITY OF LABORATORY VARIANCE

CALCULATED STATISTIC	22.531
CHI-SQUARE(.95,15)	25.000

VARIANCE IS HOMOGENEOUS
(COCHRAN'S TEST NOT PERFORMED)

DIXON'S TEST FOR LABORATORY MEANS

NONE REJECTED

LIST OF LABORATORIES WHOSE RESULTS WERE NOT USED FOR ONE-WAY ANOVA

ALL RESULTS WERE USED EXCEPT INDIVIDUAL REPLICATES LISTED BELOW

LIST OF INDIVIDUAL OUTLIERS DETECTED BY DIXON'S TEST WITHIN-LABORATORIES

NONE REJECTED

ISO/TC 102/SC 2/WG 178 - FE-TOTAL AG REDUCTION ACID DISSOLUTION
 SAMPLE: 83-2

ONE-WAY ANALYSIS OF VARIANCE TABLE

<u>SOURCE OF VARIATION</u>	<u>D.F.</u>	<u>SUM OF SQUARES</u>	<u>MEAN SQUARE</u>
BETWEEN LABS	16	.29992673E+00	.18745421E-01
WITHIN LABS	34	.15785109E+00	.46426790E-02
TOTAL	50	.45777782E+00	

REPEATABILITY VARIANCE	.46426790E-02
REPEATABILITY STD. DEV.	.06813721

BETWEEN-LABORATORIES VARIANCE	.47009139E-02
BETWEEN-LABORATORIES STD. DEV.	.06856321

REPEATABILITY	.19282830
PERMISSIBLE TOLERANCE BETWEEN LABS	.23715085

GRAND MEAN	66.81438824
VARIANCE OF THE GRAND MEAN	.36755727E-03

ISO/TC 102/SC 2/WG 17B - FE-TOTAL AG REDUCTION ACID DISSOLUTION
 SAMPLE: 76-12

LABORATORY			BOTTLE			ANALYTICAL RESULTS			LAB	LAB
NO.	COUNTRY	NO.							MEAN	VARIANCE
1	AU 1	1	60.7000	60.7100	60.7800				60.73000	.1900E-02
2	AU 2	1	60.6000	60.5000	60.5700				60.55667	.2633E-02
3	CND 1	1	60.5200	60.7100	60.5100				60.58000	.1270E-01
4	FR 1	1	60.9792	60.9060	60.7792				60.88813	.1024E-01
5	FR 2	1	60.7200	60.6000	60.7800				60.70000	.8400E-02
6	FR 3	1	60.8700	60.8500	60.7000				60.80667	.8633E-02
7	FR 4	1	60.7800	60.6900	60.8400				60.77000	.5700E-02
8	JP 1	1	60.5570	60.7270	60.7750				60.68633	.1312E-01
9	JP 2	1	60.5040	60.5970	60.5970				60.56600	.2883E-02
10	JP 3	1	60.6080	60.5440	60.5530				60.56833	.1200E-02
11	JP 4	1	60.5410	60.6340	60.5970				60.59067	.2192E-02
12	JP 5	1	60.6800	60.6500	60.6200				60.65000	.9000E-03
13	JP 6	1	60.6190	60.6100	60.6410				60.62333	.2543E-03
14	JP 7	1	60.6250	60.6620	60.6860				60.65767	.9443E-03
15	SW 1	1	60.7300	60.6300	60.6300				60.66333	.3333E-02
16	SW 2	1	60.6950	60.7540	60.5580				60.66900	.1011E-01
17	SW 3	1	60.3600	60.0500	60.4800				60.29667	.4923E-01

ISO/TC 102/SC 2/WG 17B - FE-TOTAL AG REDUCTION ACID DISSOLUTION
SAMPLE: 76-12

BARTLETT'S TEST FOR HOMOGENEITY OF LABORATORY VARIANCE

CALCULATED STATISTIC	20.674
CHI-SQUARE(.95,16)	26.301

VARIANCE IS HOMOGENEOUS
(COCHRAN'S TEST NOT PERFORMED)

DIXON'S TEST FOR LABORATORY MEANS

LAB-17 R = .569 D-CRIT(17) = .530

LIST OF LABORATORIES WHOSE RESULTS WERE NOT USED FOR ONE-WAY ANOVA

LAB-17 DIXON'S TEST FOR LABORATORY MEANS

LIST OF INDIVIDUAL OUTLIERS DETECTED BY DIXON'S TEST WITHIN-LABORATORIES

NONE REJECTED

ISO/TC 102/SC 2/WG 17B - FE-TOTAL AG REDUCTION ACID DISSOLUTION
 SAMPLE: 76-12

ONE-WAY ANALYSIS OF VARIANCE TABLE

<u>SOURCE OF VARIATION</u>	<u>D.F.</u>	<u>SUM OF SQUARES</u>	<u>MEAN SQUARE</u>
BETWEEN LABS	15	.39654107E+00	.26436071E-01
WITHIN LABS	32	.17029216E+00	.53216300E-02
TOTAL	47	.56683323E+00	

REPEATABILITY VARIANCE	.53216300E-02
REPEATABILITY STD. DEV.	.07294950

BETWEEN-LABORATORIES VARIANCE	.70381471E-02
BETWEEN-LABORATORIES STD. DEV.	.08389367

REPEATABILITY	.20644709
PERMISSIBLE TOLERANCE BETWEEN LABS	.27870776

GRAND MEAN	60.66913333
VARIANCE OF THE GRAND MEAN	.55075149E-03

ISO/TC 102/SC 2/WG 17B - FE-TOTAL AG REDUCTION ACID DISSOLUTION
SAMPLE: 81-2

LABORATORY		BOTTLE	ANALYTICAL RESULTS			LAB	LAB
NO.	COUNTRY	NO.				MEAN	VARIANCE
1	AU 1	1	59.6300	59.7200	59.7800	59.71000	.5700E-02
2	AU 2	1	59.5700	59.5800	59.6100	59.58667	.4333E-03
3	CND 1	1	59.5400	59.6100	59.7300	59.62667	.9233E-02
4	FR 1	1	59.8805	59.8988	59.7941	59.85780	.3127E-02
5	FR 2	1	59.6100	59.6100	59.6700	59.63000	.1200E-02
6	FR 3	1	59.9200	59.8800	59.8400	59.88000	.1600E-02
7	FR 4	1	59.7200	59.6700	59.8500	59.74667	.8633E-02
8	JP 1	1	59.6200	59.3990	59.6510	59.55667	.1888E-01
9	JP 2	1	59.5730	59.5730	59.5910	59.57900	.1080E-03
10	JP 3	1	59.5420	59.5180	59.5570	59.53900	.3870E-03
11	JP 4	1	59.5360	59.5730	59.5360	59.54833	.4563E-03
12	JP 5	1	59.5300	59.6500	59.5500	59.57667	.4133E-02
13	JP 6	1	59.5830	59.5460	59.6100	59.57967	.1032E-02
14	JP 7	1	59.6190	59.5650	59.5920	59.59200	.7290E-03
15	SW 1	1	59.5200	59.5200	59.6200	59.55333	.3333E-02
16	SW 2	1	59.5630	59.7040	59.5570	59.60800	.6921E-02
17	SW 3	1	59.4600	60.0000	59.8200	59.76000	.7560E-01

ISO/TC 102/SC 2/WG 17B - FE-TOTAL AG REDUCTION ACID DISSOLUTION
SAMPLE: 81-2

BARTLETT'S TEST FOR HOMOGENEITY OF LABORATORY VARIANCE

CALCULATED STATISTIC	36.334
CHI-SQUARE(.95,16)	26.301
VARIANCE IS INHOMOGENEOUS	

COCHRAN'S TEST FOR LABORATORY VARIANCE

LAB-17 C-CALC = .534 C-CRIT(3,17) = .305

DIXON'S TEST FOR LABORATORY MEANS

NONE REJECTED

LIST OF LABORATORIES WHOSE RESULTS WERE NOT USED FOR ONE-WAY ANOVA

LAB-17 COCHRAN'S TEST FOR LABORATORY VARIANCE

LIST OF INDIVIDUAL OUTLIERS DETECTED BY DIXON'S TEST WITHIN-LABORATORIES

NONE REJECTED

ISO/TC 102/SC 2/WG 17B - FE-TOTAL AG REDUCTION ACID DISSOLUTION
SAMPLE: 81-2

ONE-WAY ANALYSIS OF VARIANCE TABLE

<u>SOURCE OF VARIATION</u>	<u>D.F.</u>	<u>SUM OF SQUARES</u>	<u>MEAN SQUARE</u>
BETWEEN LABS	15	.51568265E+00	.34378843E-01
WITHIN LABS	32	.13182332E+00	.41194786E-02
TOTAL	47	.64750597E+00	

REPEATABILITY VARIANCE	.41194786E-02
REPEATABILITY STD. DEV.	.06418316

BETWEEN-LABORATORIES VARIANCE	.10086455E-01
BETWEEN-LABORATORIES STD. DEV.	.10043134

REPEATABILITY	.18163836
PERMISSIBLE TOLERANCE BETWEEN LABS	.31189366

GRAND MEAN	59.63565417
VARIANCE OF THE GRAND MEAN	.71622590E-03

ISO/TC 102/SC 2/WG 17B - FE-TOTAL AG REDUCTION ACID DISSOLUTION
 SAMPLE: 77-8

LABORATORY			BOTTLE			ANALYTICAL RESULTS			LAB	
NO.	COUNTRY	NO.							MEAN	VARIANCE
1	AU 1	1	46.1000	46.0100	45.9200				46.01000	.8100E-02
2	AU 2	1	45.8900	45.9000	45.9300				45.90667	.4333E-03
3	CND 1	1	45.8300	46.1700	46.0600				46.02000	.3010E-01
4	FR 1	1	46.0942	46.1443	46.1781				46.13887	.1782E-02
5	FR 2	1	45.9800	45.8900	46.0800				45.98333	.9033E-02
6	FR 3	1	46.0200	46.0200	46.0500				46.03000	.3000E-03
7	FR 4	1	45.8400	45.7000	46.0800				45.87333	.3693E-01
8	JP 1	1	45.8500	45.8320	45.9070				45.86300	.1533E-02
9	JP 2	1	45.7970	45.7970	45.7820				45.79200	.7500E-04
10	JP 3	1	45.9360	45.8810	45.9130				45.91000	.7630E-03
11	JP 4	1	45.7410	45.7970	45.7970				45.77833	.1045E-02
12	JP 5	1	45.9700	46.0400	45.9200				45.97667	.3633E-02
13	JP 6	1	45.8690	45.8880	45.9230				45.89333	.7503E-03
14	SW 1	1	45.9000	45.9000	45.9900				45.93000	.2700E-02
15	SW 3	1	45.8700	45.9200	45.6300				45.80667	.2403E-01

ISO/TC 102/SC 2/WG 178 - FE-TOTAL AG REDUCTION ACID DISSOLUTION
SAMPLE: 77-8

BARTLETT'S TEST FOR HOMOGENEITY OF LABORATORY VARIANCE

CALCULATED STATISTIC	31.776
CHI-SQUARE(.95,14)	23.691

VARIANCE IS INHOMOGENEOUS

COCHRAN'S TEST FOR LABORATORY VARIANCE

NONE REJECTED

DIXON'S TEST FOR LABORATORY MEANS

NONE REJECTED

LIST OF LABORATORIES WHOSE RESULTS WERE NOT USED FOR ONE-WAY ANOVA

ALL RESULTS WERE USED EXCEPT INDIVIDUAL REPLICATES LISTED BELOW

LIST OF INDIVIDUAL OUTLIERS DETECTED BY DIXON'S TEST WITHIN-LABORATORIES

NONE REJECTED

ISO/TC 102/SC 2/WG 17B - FE-TOTAL AG REDUCTION ACID DISSOLUTION
 SAMPLE: 77-8

ONE-WAY ANALYSIS OF VARIANCE TABLE

<u>SOURCE OF VARIATION</u>	<u>D.F.</u>	<u>SUM OF SQUARES</u>	<u>MEAN SQUARE</u>
BETWEEN LABS	14	.42089742E+00	.30064102E-01
WITHIN LABS	30	.24243055E+00	.80810185E-02
TOTAL	44	.66332798E+00	

REPEATABILITY VARIANCE	.80810185E-02
REPEATABILITY STD. DEV.	.08989449

BETWEEN-LABORATORIES VARIANCE	.73276944E-02
BETWEEN-LABORATORIES STD. DEV.	.08560195

REPEATABILITY	.25440139
PERMISSIBLE TOLERANCE BETWEEN LABS	.30173963

GRAND MEAN	45.92748000
VARIANCE OF THE GRAND MEAN	.66809115E-03

ISO/TC 102/SC 2/WG 23B

Fe-total - TiCl_3 reduction - Perchloric acid reoxidation

APPENDIX A3

DETAILED COMPUTATION OF ANALYTICAL DATA

ISO/TC 102/SC 2/WG 23A - TOTAL IRON BY POLLUTION FREE TICL3 METHOD
 SAMPLE 1: 76-17

LABORATORY			BOTTLE			ANALYTICAL RESULTS			LAB	LAB
NO.	COUNTRY	NO.							MEAN	VARIANCE
1	AU 1	1	67.0500	66.9900	67.1000				67.04667	.3033E-02
2	AU 2	1	67.0620	67.1720	67.0870				67.10700	.3325E-02
3	AU 3	1	67.2700	67.1900	67.2400				67.23333	.1633E-02
4	AU 4	1	66.9200	66.9000	66.9800				66.93333	.1733E-02
5	BR 1	1	66.9300	67.0900	67.1700				67.06333	.1493E-01
6	BR 2	1	67.1600	67.3400	67.3600				67.28667	.1213E-01
7	BR 3	1	67.3700	66.9500					67.16000	.8820E-01
8	CND 1	1	67.1900	67.1470	67.1310				67.15600	.9310E-03
9	CND 2	1	67.2540	67.2110	67.2510				67.23133	.2029E-02
			67.1880	67.1840	67.3000					
10	CND 3	1	67.3200	67.2000	67.1200				67.21333	.1013E-01
11	FR 1	1	67.3190	66.9660	67.0630				67.11600	.3326E-01
12	FR 2	1	67.5100	67.5800	67.6300				67.57333	.3633E-02
13	FR 3	1	66.7290	66.7750	66.8030				66.76900	.1396E-02
14	FR 4	1	67.3524	67.1825	67.1753				67.23673	.1005E-01
15	FR 5	1	67.3800	67.3800	67.3800				67.38000	0.
16	FR 6	1	67.4700	67.5900	67.5600				67.54000	.3900E-02
17	GER 1	1	67.1700	67.1200	67.1200				67.13667	.8333E-03
18	GER 2	1	67.0561	67.0674	66.9184				67.02623	.3120E-02
			67.0569	67.0439	67.0147					
19	GER 3	1	67.5700	67.3900	68.2700				67.74333	.2161E+00
20	IT 1	1	67.3700	67.3100	67.4000				67.36000	.2100E-02
21	IT 2	1	67.5000	67.3600	67.4000				67.42000	.5200E-02
22	JP 1	1	66.9800	66.8900	66.8500				66.90667	.4433E-02
23	JP 2	1	67.1100	66.9700	67.1300				67.07000	.7600E-02
24	JP 3	1	67.1300	67.1600	67.1400				67.14333	.2333E-03
25	JP 4	1	67.2010	67.1930	67.1820				67.19200	.9100E-04
26	JP 5	1	67.0650	67.0640	67.0660				67.06500	.1000E-05
27	SA 1	1	67.1600	67.1500	67.2600				67.19000	.3700E-02
28	SA 2	1	67.0800	67.1300	67.1500				67.12000	.1300E-02
29	SW 1	1	66.9500	67.1600	67.0200				67.04333	.1143E-01
30	USA 1	1	67.2390	67.1770	67.3590				67.31833	.1287E-01
			67.3870	67.2610	67.4870					
31	USA 2	1	67.1270	67.0050					67.01600	.2420E-03
32	USA 3	1	67.1400	67.4800	67.5100				67.44333	.8233E-02
33	USA 4	1	67.1020	67.3030	67.2570				67.27250	.1814E-01
			67.4280							
34	USA 5	1	67.1250	67.0350	67.0920				67.08400	.2073E-02

ISO/TC 102/SC 2/WG 23A - TOTAL IRON BY POLLUTION FREE TICL3 METHOD
SAMPLE 1: 76-17

BARTLETT'S TEST FOR HOMOGENEITY OF LABORATORY VARIANCE

CALCULATED STATISTIC	87.383
CHI-SQUARE(.95,32)	46.201

VARIANCE IS INHOMOGENEOUS

COCHRAN'S TEST FOR LABORATORY VARIANCE

LAB-19	C-CALC = .443	C-CRIT(3,33) = .184
LAB- 7	C-CALC = .324	C-CRIT(3,32) = .188

DIXON'S TEST FOR LABORATORY MEANS

NONE REJECTED

LIST OF LABORATORIES WHOSE RESULTS WERE NOT USED FOR ONE-WAY ANOVA

LAB- 7	COCHRAN'S TEST FOR LABORATORY VARIANCE
LAB-19	COCHRAN'S TEST FOR LABORATORY VARIANCE

LIST OF INDIVIDUAL OUTLIERS DETECTED BY DIXON'S TEST WITHIN-LABORATORIES

NONE REJECTED

ISO/TC 102/SC 2/WG 23A - TOTAL IRON BY POLLUTION FREE TICL3 METHOD
 SAMPLE 1: 76-17

ONE-WAY ANALYSIS OF VARIANCE TABLE

<u>SOURCE OF VARIATION</u>	<u>D.F.</u>	<u>SUM OF SQUARES</u>	<u>MEAN SQUARE</u>
BETWEEN LABS	31	.30853285E+01	.99526725E-01
WITHIN LABS	73	.43943007E+00	.60195900E-02
TOTAL	104	.35247585E+01	

REPEATABILITY VARIANCE	.60195900E-02
REPEATABILITY STD. DEV.	.07758602

BETWEEN-LABORATORIES VARIANCE	.28568211E-01
BETWEEN-LABORATORIES STD. DEV.	.16902133

REPEATABILITY	.21956843
PERMISSIBLE TOLERANCE BETWEEN LABS	.50289670

GRAND MEAN	67.18156762
VARIANCE OF THE GRAND MEAN	.10186724E-02

ISO/TC 102/SC 2/WG 23A - TOTAL IRON BY POLLUTION FREE TICL3 METHOD
SAMPLE 2: 83-2

LABORATORY			BOTTLE			ANALYTICAL RESULTS			LAB	LAB
NO.	COUNTRY	NO.							MEAN	VARIANCE
1	AU 1	1	66.8500	66.8400	66.8700				66.85333	.2333E-03
2	AU 2	1	66.6570	66.7820	66.6890				66.70933	.4216E-02
3	AU 3	1	66.7800	66.7200	66.7400				66.74667	.9333E-03
4	AU 4	1	66.6600	66.6800	66.6900				66.67667	.2333E-03
5	BR 1	1	66.4700	66.6600	66.6300				66.58667	.1043E-01
6	BR 2	1	66.8000	66.8200	66.8000				66.80667	.1333E-03
7	BR 3	1	66.8000	66.9700					66.88500	.1445E-01
8	CND 1	1	66.8340	66.8590	66.7620				66.81833	.2536E-02
9	CND 2	1	66.8290	66.7280	66.7180				66.74500	.4323E-02
			66.7860	66.6390	66.7700					
10	CND 3	1	66.7300	66.7100	66.6100				66.68333	.4133E-02
11	FR 1	1	66.8430	66.6470	66.6540				66.71467	.1236E-01
12	FR 2	1	66.8000	67.1800	66.7900				66.92333	.4943E-01
13	FR 3	1	67.0090	66.5790	66.9970				66.86167	.5996E-01
14	FR 4	1	66.8178	66.7265	66.8009				66.78173	.2359E-02
15	FR 5	1	66.8100	66.8100	66.8100				66.81000	.2910E-10
16	FR 6	1	66.8800	66.8600	66.8600				66.86667	.1333E-03
17	GER 1	1	66.8300	66.7300	66.7800				66.78000	.2500E-02
18	GER 2	1	66.4367	66.5510	66.6059				66.56120	.4200E-02
			66.6041	66.5698	66.5997					
19	GER 3	1	66.9500	66.4300	67.0300				66.80333	.1061E+00
20	IT 1	1	66.6600	66.8000	66.7500				66.73667	.5033E-02
21	IT 2	1	66.7900	66.8700	66.9200				66.86000	.4300E-02
22	JP 1	1	66.5200	66.5500	66.4800				66.51667	.1233E-02
23	JP 2	1	66.7500	66.8000	66.7400				66.76333	.1033E-02
24	JP 3	1	66.8300	66.8100	66.8500				66.83000	.4000E-03
25	JP 4	1	66.7940	66.7600	66.7550				66.76967	.4503E-03
26	JP 5	1	66.8630	66.8380	66.8450				66.84867	.1663E-03
27	SA 1	1	66.9000	66.8600	66.8500				66.87000	.7000E-03
28	SA 1A	1	66.8100	66.6100	67.0800				66.83333	.5563E-01
29	SA 2	1	66.8500	66.7700	66.8600				66.82667	.2433E-02
30	SW 1	1	66.2500	66.3200	66.4600				66.34333	.1143E-01
31	USA 1	1	66.7450	66.7680	66.6890				66.82883	.1160E-01
			66.9410	66.9030	66.9270					
32	USA 2	1	66.6090	66.5710					66.59000	.7220E-03
33	USA 3	1	66.7900	66.7100	66.7300				66.74333	.1733E-02
34	USA 4	1	66.5810	66.6230	66.6040				66.61300	.7220E-03
			66.6440							
35	USA 5	1	66.8230	66.7010	66.6830				66.73567	.5801E-02

ISO/TC 102/SC 2/WG 23A - TOTAL IRON BY POLLUTION FREE TICL3 METHOD
 SAMPLE 2: 83-2

BARTLETT'S TEST FOR HOMOGENEITY OF LABORATORY VARIANCE

CALCULATED STATISTIC	118.542
CHI-SQUARE(.95,34)	48.611

VARIANCE IS INHOMOGENEOUS

COCHRAN'S TEST FOR LABORATORY VARIANCE

LAB-19	C-CALC = .278	C-CRIT(3,35) = .175
LAB-13	C-CALC = .217	C-CRIT(3,34) = .179
LAB-28	C-CALC = .258	C-CRIT(3,33) = .184
LAB-12	C-CALC = .308	C-CRIT(3,32) = .188

DIXON'S TEST FOR LABORATORY MEANS

LAB-30	R = .414	D-CRIT(35) = .389
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LIST OF LABORATORIES WHOSE RESULTS WERE NOT USED FOR ONE-WAY ANOVA

LAB-12	COCHRAN'S TEST FOR LABORATORY VARIANCE
LAB-13	COCHRAN'S TEST FOR LABORATORY VARIANCE
LAB-19	COCHRAN'S TEST FOR LABORATORY VARIANCE
LAB-28	COCHRAN'S TEST FOR LABORATORY VARIANCE
LAB-30	DIXON'S TEST FOR LABORATORY MEANS

LIST OF INDIVIDUAL OUTLIERS DETECTED BY DIXON'S TEST WITHIN-LABORATORIES

NONE REJECTED

ISO/TC 102/SC 2/WG 23A - TOTAL IRON BY POLLUTION FREE TICL3 METHOD
SAMPLE 2: 83-2

ONE-WAY ANALYSIS OF VARIANCE TABLE

<u>SOURCE OF VARIATION</u>	<u>D.F.</u>	<u>SUM OF SQUARES</u>	<u>MEAN SQUARE</u>
BETWEEN LABS	29	.95633414E+00	.32977039E-01
WITHIN LABS	68	.24494272E+00	.36020988E-02
TOTAL	97	.12012769E+01	

REPEATABILITY VARIANCE	.36020988E-02
REPEATABILITY STD. DEV.	.06001749

BETWEEN-LABORATORIES VARIANCE	.90194016E-02
BETWEEN-LABORATORIES STD. DEV.	.09497053

REPEATABILITY	.16984949
PERMISSIBLE TOLERANCE BETWEEN LABS	.29438055

GRAND MEAN	66.74710612
VARIANCE OF THE GRAND MEAN	.36357324E-03

ISO/TC 102/SC 2/WG 23A - TOTAL IRON BY POLLUTION FREE TICL3 METHOD
 SAMPLE 3: 76-12

LABORATORY			ANALYTICAL RESULTS			LAB	LAB
NO.	COUNTRY	BOTTLE NO.				MEAN	VARIANCE
1	AU 1	1	60.7900	60.7100	60.7300	60.74333	.1733E-02
2	AU 2	1	60.5970	60.6490	60.5890	60.61167	.1061E-02
3	AU 3	1	60.6800	60.7400	60.7300	60.71667	.1033E-02
4	AU 4	1	60.5100	60.4700	60.4800	60.48667	.4333E-03
5	BR 1	1	60.5600	60.6900	60.6300	60.62667	.4233E-02
6	BR 2	1	60.5700	60.5800	60.5800	60.57667	.3333E-04
7	BR 3	1	60.3400	60.9500		60.64500	.1860E+00
8	CND 1	1	60.6040	60.6070	60.7400	60.65033	.6032E-02
9	CND 2	1	60.6920	60.5910	60.6550	60.64233	.1302E-02
			60.6470	60.6580	60.6110		
10	CND 3	1	60.7400	60.7300	60.7400	60.73667	.3333E-04
11	FR 1	1	60.5620	60.5940	60.9240	60.69333	.4016E-01
12	FR 2	1	60.6900	60.6800	60.6900	60.68667	.3333E-04
13	FR 3	1	60.6140	60.8920	60.7290	60.74500	.1951E-01
14	FR 4	1	60.6917	60.8033	60.8233	60.77277	.5029E-02
15	FR 5	1	60.8100	60.7400	60.8100	60.78667	.1633E-02
16	FR 6	1	60.6000	60.7900	60.5900	60.66000	.1270E-01
17	GER 1	1	60.5800	60.7300	60.6500	60.65333	.5633E-02
18	GER 2	1	60.5838	60.7905	60.5745	60.62218	.9208E-02
			60.5386	60.6819	60.5638		
19	GER 3	1	60.7200	60.6500	60.5400	60.63667	.8233E-02
20	IT 1	1	60.5800	60.7000	60.6600	60.64667	.3733E-02
21	IT 2	1	60.7000	60.8000	60.7500	60.75000	.2500E-02
22	JP 1	1	60.4000	60.3500	60.6100	60.45333	.1903E-01
23	JP 2	1	60.6800	60.5100	60.6800	60.62333	.9633E-02
24	JP 3	1	60.7000	60.7000	60.8100	60.73667	.4033E-02
25	JP 4	1	60.6320	60.6560	60.6120	60.63333	.4853E-03
26	JP 5	1	60.5660	60.6460	60.6090	60.60700	.1603E-02
27	SA 1	1	60.6500	60.8000	60.8600	60.77000	.1170E-01
28	SA 1A	1	60.5100	60.5600	60.6800	60.58333	.7633E-02
29	SA 2	1	60.7600	60.7900	60.8100	60.78667	.6333E-03
30	SW 1	1	60.4600	60.3200	60.7400	60.50667	.4573E-01
31	USA 1	1	60.7260	60.7930	60.5880	60.77567	.2758E-01
			61.0700	60.8110	60.6660		
32	USA 2	1	60.6380	60.6510		60.64450	.8450E-04
33	USA 3	1	60.6000	60.6900	60.9700	60.75333	.3723E-01
34	USA 4	1	60.8790	60.5860	60.5490	60.69250	.2358E-01
			60.7560				
35	USA 5	1	60.5980	60.6840	60.7040	60.66200	.3172E-02

ISO/TC 102/SC 2/WG 23A - TOTAL IRON BY POLLUTION FREE TICL3 METHOD
SAMPLE 3: 76-12

BARTLETT'S TEST FOR HOMOGENEITY OF LABORATORY VARIANCE

CALCULATED STATISTIC	81.879
CHI-SQUARE(.95,34)	48.611

VARIANCE IS INHOMOGENEOUS

COCHRAN'S TEST FOR LABORATORY VARIANCE

LAB- 7 C-CALC = .370 C-CRIT(3,35) = .175

DIXON'S TEST FOR LABORATORY MEANS

NONE REJECTED

LIST OF LABORATORIES WHOSE RESULTS WERE NOT USED FOR ONE-WAY ANOVA

LAB- 7 COCHRAN'S TEST FOR LABORATORY VARIANCE

LIST OF INDIVIDUAL OUTLIERS DETECTED BY DIXON'S TEST WITHIN-LABORATORIES

NONE REJECTED

ISO/TC 102/SC 2/WG 23A - TOTAL IRON BY POLLUTION FREE TICL3 METHOD
 SAMPLE 3: 76-12

ONE-WAY ANALYSIS OF VARIANCE TABLE

<u>SOURCE OF VARIATION</u>	<u>D.F.</u>	<u>SUM OF SQUARES</u>	<u>MEAN SQUARE</u>
BETWEEN LABS	33	.75651806E+00	.22924790E-01
WITHIN LABS	77	.77059089E+00	.10007674E-01
TOTAL	110	.15271090E+01	

REPEATABILITY VARIANCE	.10007674E-01
REPEATABILITY STD. DEV.	.10003836

BETWEEN-LABORATORIES VARIANCE	.39654203E-02
BETWEEN-LABORATORIES STD. DEV.	.06297158

REPEATABILITY	.28310857
PERMISSIBLE TOLERANCE BETWEEN LABS	.26801844

GRAND MEAN	60.66834595
VARIANCE OF THE GRAND MEAN	.21535592E-03

ISO/TC 102/SC 2/WG 23A - TOTAL IRON BY POLLUTION FREE TICL3 METHOD
SAMPLE 4: 81-2

LABORATORY			ANALYTICAL RESULTS			LAB	LAB
NO.	COUNTRY	BOTTLE NO.				MEAN	VARIANCE
1	AU 1	1	59.6200	59.6600	59.6400	59.64000	.4000E-03
2	AU 2	1	59.6030	59.6560	59.5460	59.60167	.3026E-02
3	AU 3	1	59.5900	59.5700	59.5500	59.57000	.4000E-03
4	AU 4	1	59.6200	59.5500	59.5600	59.57667	.1433E-02
5	BR 1	1	59.4000	59.5600	59.5100	59.49000	.6700E-02
6	BR 2	1	59.5600	59.4700	59.5200	59.51667	.2033E-02
7	BR 3	1	59.7300	59.8500		59.79000	.7200E-02
8	CND 1	1	59.5070	59.5010	59.5200	59.50933	.9433E-04
9	CND 2	1	59.5610	59.6240	59.6810	59.62100	.2765E-02
			59.5630	59.6190	59.6780		
10	CND 3	1	59.4800	59.4500	59.4300	59.45333	.6333E-03
11	FR 1	1	59.5770	59.5610	59.6610	59.59967	.2885E-02
12	FR 2	1	59.4600	59.6400	59.5300	59.54333	.8233E-02
13	FR 3	1	59.2830	59.4910	59.4760	59.41667	.1346E-01
14	FR 4	1	59.5898	59.6509	59.6436	59.62810	.1113E-02
15	FR 5	1	59.6800	59.6500	59.6800	59.67000	.3000E-03
16	FR 6	1	59.4200	59.5300	59.5100	59.48667	.3433E-02
17	GER 1	1	59.5300	59.5300	59.6000	59.55333	.1633E-02
18	GER 2	1	59.4775	59.5249	59.4616	59.47837	.1033E-02
			59.4855	59.4286	59.4921		
19	GER 3	1	59.2200	59.4000	59.4500	59.35667	.1463E-01
20	IT 1	1	59.5500	59.5500	59.5900	59.56333	.5333E-03
21	IT 2	1	59.6400	59.7800	59.7100	59.71000	.4900E-02
22	JP 1	1	59.2800	59.4200	59.3100	59.33667	.5433E-02
23	JP 2	1	59.5700	59.5400	59.4600	59.52333	.3233E-02
24	JP 3	1	59.5900	59.6400	59.6300	59.62000	.7000E-03
25	JP 4	1	59.5170	59.5360	59.5400	59.53100	.1510E-03
26	SA 1	1	59.6200	59.6500	59.8400	59.70333	.1423E-01
27	SA 1A	1	59.5000	59.5800	59.6500	59.57667	.5633E-02
28	SA 2	1	59.7800	59.8600	59.8700	59.83667	.2433E-02
29	SW 1	1	59.4100	59.4800	59.6200	59.50333	.1143E-01
30	USA 1	1	59.5120	59.7290	59.5930	59.57917	.1348E-01
			59.3920	59.6480	59.6010		
31	USA 2	1	59.5980	59.5410		59.56950	.1625E-02
32	USA 3	1	59.6400	59.5600	59.8000	59.66667	.1493E-01
33	USA 4	1	59.5180	59.5100	59.6060	59.56225	.3128E-02
			59.6150				
34	USA 5	1	59.5530	59.6340	59.6450	59.61067	.2524E-02

ISO/TC 102/SC 2/WG 23A - TOTAL IRON BY POLLUTION FREE TICL3 METHOD
SAMPLE 4: 81-2

BARTLETT'S TEST FOR HOMOGENEITY OF LABORATORY VARIANCE

CALCULATED STATISTIC	43.330
CHI-SQUARE(.95,33)	47.407

VARIANCE IS HOMOGENEOUS
(COCHRAN'S TEST NOT PERFORMED)

DIXON'S TEST FOR LABORATORY MEANS

NONE REJECTED

LIST OF LABORATORIES WHOSE RESULTS WERE NOT USED FOR ONE-WAY ANOVA

ALL RESULTS WERE USED EXCEPT INDIVIDUAL REPLICATES LISTED BELOW

LIST OF INDIVIDUAL OUTLIERS DETECTED BY DIXON'S TEST WITHIN-LABORATORIES

NONE REJECTED

ISO/TC 102/SC 2/WG 23A - TOTAL IRON BY POLLUTION FREE TICL3 METHOD
 SAMPLE 4: 81-2

ONE-WAY ANALYSIS OF VARIANCE TABLE

<u>SOURCE OF VARIATION</u>	<u>D.F.</u>	<u>SUM OF SQUARES</u>	<u>MEAN SQUARE</u>
BETWEEN LABS	33	.10878953E+01	.32966523E-01
WITHIN LABS	76	.35769983E+00	.47065768E-02
TOTAL	109	.14455951E+01	

REPEATABILITY VARIANCE	.47065768E-02
REPEATABILITY STD. DEV.	.06860450

BETWEEN-LABORATORIES VARIANCE	.87558556E-02
BETWEEN-LABORATORIES STD. DEV.	.09357273

REPEATABILITY	.19415072
PERMISSIBLE TOLERANCE BETWEEN LABS	.29828178

GRAND MEAN	59.56745909
VARIANCE OF THE GRAND MEAN	.32065884E-03

ISO/TC 102/SC 2/WG 23A - TOTAL IRON BY POLLUTION FREE TICL3 METHOD
 SAMPLE 5: 77-8

LABORATORY			BOTTLE			ANALYTICAL RESULTS			LAB	
NO.	COUNTRY	NO.							MEAN	VARIANCE
1	AU 1	1	45.8400	45.8700	45.8800				45.86333	.4333E-03
2	AU 2	1	45.7900	45.8100	45.8420				45.81400	.6880E-03
3	AU 3	1	46.0200	45.9900	45.9300				45.98000	.2100E-02
4	AU 4	1	45.7600	45.7200	45.7100				45.73000	.7000E-03
5	BR 1	1	45.7400	45.8600	45.7500				45.78333	.4433E-02
6	BR 2	1	45.8400	45.8100	45.8300				45.82667	.2333E-03
7	BR 3	1	46.2100	45.9600					46.08500	.3125E-01
8	CND 1	1	45.8510	45.8620	45.8980				45.87033	.6043E-03
9	CND 2	1	45.9690	45.8890	45.9680				45.94383	.3085E-02
			46.0260	45.8780	45.9330					
10	CND 3	1	45.9200	45.8800	45.8700				45.89000	.7000E-03
11	FR 1	1	45.9960	45.9060	46.1740				46.02533	.1860E-01
12	FR 2	1	45.8400	46.0400	45.9800				45.95333	.1053E-01
13	FR 3	1	45.9060	45.6840	46.1040				45.89800	.4415E-01
14	FR 4	1	46.0766	46.0898	45.9761				46.04750	.3867E-02
15	FR 5	1	45.9400	45.8700	45.8700				45.89333	.1633E-02
16	FR 6	1	45.7900	45.8000	45.7900				45.79333	.3333E-04
17	GER 1	1	45.9500	46.0500	45.8800				45.96000	.7300E-02
18	GER 2	1	45.7996	45.6423	45.7392				45.73177	.5094E-02
			45.7131	45.6710	45.8254					
19	GER 3	1	45.5200	45.7000	45.9800				45.73333	.5373E-01
20	IT 1	1	45.7900	45.8100	45.8300				45.81000	.4000E-03
21	IT 2	1	46.0300	46.0500	46.0100				46.03000	.4000E-03
22	JP 1	1	45.6000	45.7100	45.6300				45.64667	.3233E-02
23	JP 2	1	45.7700	45.7800	45.8700				45.80667	.3033E-02
24	JP 3	1	45.7500	45.8200	45.8000				45.79000	.1300E-02
25	JP 4	1	45.8560	45.8660	45.8580				45.86000	.2800E-04
26	SA 1	1	45.9200	45.9900	46.0400				45.98333	.3633E-02
27	SA 1A	1	45.7400	45.9400	46.0700				45.91667	.2763E-01
28	SA 2	1	45.9900	45.9200	45.8900				45.93333	.2633E-02
29	SW 1	1	45.3800	45.9400	45.5200				45.61333	.8493E-01
30	USA 1	1	45.8940	45.8940	45.8740				45.90017	.5219E-02
			45.7880	45.9490	46.0020					
31	USA 2	1	45.5730	45.5860					45.57950	.8450E-04
32	USA 3	1	45.7600	45.7100	45.6600				45.71000	.2500E-02
33	USA 4	1	45.8720	45.7950	46.0850				45.91225	.1515E-01
			45.8970							
34	USA 5	1	45.8030	45.8190	45.8700				45.83067	.1224E-02

ISO/TC 102/SC 2/WG 23A - TOTAL IRON BY POLLUTION FREE TICL3 METHOD
SAMPLE 5: 77-8

BARTLETT'S TEST FOR HOMOGENEITY OF LABORATORY VARIANCE

CALCULATED STATISTIC	83.643
CHI-SQUARE(.95,33)	47.407

VARIANCE IS INHOMOGENEOUS

COCHRAN'S TEST FOR LABORATORY VARIANCE

LAB-29	C-CALC = .249	C-CRIT(3,34) = .179
LAB-19	C-CALC = .210	C-CRIT(3,33) = .184
LAB-13	C-CALC = .219	C-CRIT(3,32) = .188
LAB- 7	C-CALC = .198	C-CRIT(3,31) = .193
LAB-27	C-CALC = .218	C-CRIT(3,30) = .198

DIXON'S TEST FOR LABORATORY MEANS

NONE REJECTED

LIST OF LABORATORIES WHOSE RESULTS WERE NOT USED FOR ONE-WAY ANOVA

LAB- 7	COCHRAN'S TEST FOR LABORATORY VARIANCE
LAB-13	COCHRAN'S TEST FOR LABORATORY VARIANCE
LAB-19	COCHRAN'S TEST FOR LABORATORY VARIANCE
LAB-27	COCHRAN'S TEST FOR LABORATORY VARIANCE
LAB-29	COCHRAN'S TEST FOR LABORATORY VARIANCE

LIST OF INDIVIDUAL OUTLIERS DETECTED BY DIXON'S TEST WITHIN-LABORATORIES

NONE REJECTED

ISO/TC 102/SC 2/WG 23A - TOTAL IRON BY POLLUTION FREE TICL3 METHOD
 SAMPLE 5: 77-8

ONE-WAY ANALYSIS OF VARIANCE TABLE

<u>SOURCE OF VARIATION</u>	<u>D.F.</u>	<u>SUM OF SQUARES</u>	<u>MEAN SQUARE</u>
BETWEEN LABS	28	.10887483E+01	.38883869E-01
WITHIN LABS	67	.25301278E+00	.37763101E-02
TOTAL	95	.13417611E+01	

REPEATABILITY VARIANCE	.37763101E-02
REPEATABILITY STD. DEV.	.06145169

BETWEEN-LABORATORIES VARIANCE	.10636736E-01
BETWEEN-LABORATORIES STD. DEV.	.10313455

REPEATABILITY	.17390828
PERMISSIBLE TOLERANCE BETWEEN LABS	.31671848

GRAND MEAN	45.86204271
VARIANCE OF THE GRAND MEAN	.43636749E-03